# INVESTIGATION OF THE POTENTIAL TO DEVELOP HIGH PULP STRENGTH FROM HIGH YIELD KRAFT PULP MADE FROM *PINUS PATULA*

**By**

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## Abstract

The major focus of this study was to provide better understanding of the potential of increasing pulp yield and developing high pulp strength from high yield Kraft pulps made from *Pinus patula*. This included understanding the impact of Specific Refining Energy (SRE) on strength development and the possible ways of improving pulp strength at different yields and Kappa numbers. This study consisted of three phases. The phase one trials compared industrial and laboratory High Consistency (HC) and Low Consistency (LC) refining. The focus of this work was to establish whether the Council for Scientific and Industrial Research (CSIR) Forestry and Forest Products Research Centre (FFP) laboratory refiners can be used to simulate the industrial refining process. It was found that the laboratory HC refined samples at an SRE of 200 kWh/t followed by LC refining at 80 kWh/t produced pulps with similar pulp properties to those currently achieved at Tugela mill.

The second phase trials were aimed at developing an understanding of how the strength properties of high Kappa number pulps can be optimized. Thus the influences of Kappa number and SRE on strength development in industrial pulps were studied. The results indicated that in order to achieve optimum strength development, pulp at different Kappa numbers require different SRE's. The Pulp at Kappa number 70 reached optimum strength properties at a total SRE of 280 kWh/t (after HC and LC) while pulp at Kappa number 85 attained optimum strength at 380 kWh/t. More importantly, the evaluation on energy input per unit kappa number increase did show that an average increase of 15 units of Kappa number required an average increase of 100 units of SRE as total energy input (HC + LC). This energy input estimate was then used in optimizing the refining conditions for phase three trials.

Phase three trials investigated the influence of Kappa number on total pulp yield as well as potential of developing high strength from high yield Kraft pulp at Kappa number 100, 115 and 130. The pulping results indicated an average yield increase of 1.6% for every 15 units of Kappa number increase. However, the refining results indicated that improving pulp yield of *Pinus patula* through conventional Kraft pulping (cooked at temperature of 170 °C, liquor charge: 16% and sulphidity: 32%) by increasing Kappa number had a negative effect on pulp strength properties.

From the overall experimental results, a sufficient understanding of the effect of Kappa number, pulping method and SRE on fibre strength development was achieved. This led to a recommendation of modified pulping and refining for future studies.

## Preface

I, the undersigned, hereby declare that unless specifically stated to the contrary in the text, the work contained in this thesis is my original work and has not been either submitted in part or in whole to any other university. The research work described in this thesis was carried out at the University of KwaZulu-Natal at the Forestry and Forest Products Research Centre of the CSIR in Durban and was supervised by Mr. Iain Kerr and Dr Valerie Grzeskowiak.

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J.K. Johakimu

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## Abbreviation

| SYMBOL | DESCRIPTION | UNITS |
|---|---|---|
| **APMP** | Alkaline peroxide mechanical pulping | - |
| AQ | Anthraquinone | - |
| **EA** | Effective alkali refers to 1/2[$Na_2S$] and [NaOH] in cooking liquor | **g/l** |
| **°C** | Temperature in degrees centigrade | - |
| **CEL** | Cutting edge length | rev/min |
| **CosF** | Phase angle | - |
| **DBH** | Diameter at Breast Height | cm |
| **DP** | Degree of polymerization of the cellulose chain | - |
| **Ea** | Activation energy | kcal/mol |
| **HC** | High consistency | % |
| **HCR** | High Consistency Refiner | - |
| **H-factor** | The relative reaction rate at a given temperature and time is plotted vs. the cooking time, the area under the curves gives a single numerical value called the H-factor | - |
| **HYK** | High yield Kraft pulping process | - |
| **HYS** | High yield sulphite pulping process | - |
| **$I_o$** | No load current | Amps |
| **$I_t$** | Gross current | Amps |
| **K** | Temperature in Kelvin | - |
| **$K_r$** | Reaction rate constant | - |
| **LC** | Low consistency | % |
| **LCR** | Low Consistency Refiner | - |
| **$M_1$** | Mass of pulp suspension | kg |
| **Mo** | Mass of wet pulp | kg |
| **N** | Number of bars | - |
| **NSSC** | Neutral sulphite semi chemical pulping process | - |
| **PS** | Digester additive (Polysulphide) | - |
| **$P_t$** | Gross power | kW |
| **$P_o$** | No load power | kW |
| **$P_{net}$** | Net power (Gross power – No load power) | kW |
| **r** | Radius of the refining zone | m |
| **RI** | Refining intensity (expressed as average specific energy per impact) | J/kg |
| **SRE** | Specific refining energy | kWh/t |

| | | |
|---|---|---|
| **STD** | Standard deviation | - |
| **TEA** | Tensile energy absorption | kJ/kg |
| $\mu_t$ | Tangential coefficients | - |
| $\mu r$ | Radial friction coefficients | - |
| **V** | Voltage | Watts |
| $X_i$ | Pulp consistency of wet pulp | % |
| $X_o$ | Pulp consistency of wet pulp | % |
| $\omega$ | Refiner rotational speed | rev/sec |

# CHAPTER 1: INTRODUCTION

Due to the unpredictable trend of rising costs of raw materials, tight environmental regulations and the current impact of trade liberalization policy, fibre yield optimisation remains a priority in the present pulp and paper market. As this approach reduces the costs of purchasing wood, thus improves mill competitiveness either in local or global market (McGovern, 1972; Helle, 1998; Venter, 2005).

In this regard, many approaches have been considered to produce High Yield Kraft (HYK) pulp (McGovern, 1972; Kleppe and Storebraten, 1985; Jarkko, 2005). These include pulping to a higher Kappa number. Kappa number is the measure of the degree of delignification (how much amount of lignin left in the pulp mass), the higher the Kappa the high the lignin content and also the higher the yield. Yield is the measure of the amount of wood converted into pulp per given wood chips charged into the digester, thus defines the economical value of the pulping process. Benefits of pulping to higher Kappa number include: 1) reduction in wood input, which enhances mill profitability through reducing purchasing costs for the wood, and 2) improved energy performance as it uses less energy in the digester to produce one ton of pulp.

It is obvious that high yield pulps are rich in lignin which is hydrophobic in nature (Christensen, 1998; Helle, 1998). It is well accepted that a high amount of lignin leads to a decrease in strength properties. These drawbacks limit the application of high yield pulps in paper making furnishes. Thus, finding ways of improving pulp strength from high HYK pulps are of paramount importance to the paper industry.

To address this challenge most of the research and development work (Kleppe, 1970; Christensen, 1998; Jarkko, 2005) has focused on modifying both pulping and refining techniques to improve the degree of hydrophilicity of lignin present in high yield pulps. In this study, the potential to develop high strength from HYK pulps based on conventional Kraft pulping, followed by high consistency and low consistency refining, was studied. The focus was to provide a better understanding of the influence of Kappa number on pulp yield and the influence of refining techniques on the potential of developing high strength from HYK pulps. The information obtained in this study could be used by the paper industry either to adjust pulping and refining conditions, or to define the most appropriate application of HYK pulps.

## 1.1 Hypothesis

HYK pulp produced at Kappa number higher than 70 if suitably treated using high consisteny refining (HCR) will achieve the desired strength properties required for sack and linerboard Kraft grades.

## 1.2 Aims and objectives

The work described in this study had three specific objectives:

1) Simulation of industrial refining: This was done by comparing refining results using industrial and laboratory high consistency (HC) and low consistency (LC) refiners. The focus of this work was to establish whether FFP (Forestry and Forest Products Research Centre) laboratory refiners can be used to simulate industrial refining processes. This was critical for the subsequent work in this project.
2) Establishing if and how the strength properties of high Kappa number pulps can be optimized: This was done by investigating the effect of Kappa number increase and specific refining energy (SRE) on pulp strength at Kappa 70 and 85 for industrial pulp. An understanding of these effects was essential in the setting of the refining conditions to optimize pulp strength properties at high Kappa numbers.
3) Investigation on the influence of Kappa number on total pulp yield and the potential of developing high strength properties from high yield Kraft pulps: This was done by producing pulp at Kappa numbers 100, 115 and 130 by the conventional Kraft pulping method, followed by HC and LC refining. The focus was to establish whether the yield gain and strength properties will provide an economic benefit to production of HYK pulps, in particular for manufacturing sack paper grade.

# CHAPTER 2: LITERATURE REVIEW

## 2.1 Introduction

The literature review covers two important aspects. The first aspect of the study reviews on Kraft pulping technology, the most emphasis being on the recent developments in high yield Kraft pulping processes and theories of improving yield and pulp strength. This includes also understanding the influence of raw material quality and the effect of Kraft pulping variables. The second aspect of the study reviews on fibre strength development during refining. These include the effect of refining variables, plate pattern and materials and refining techniques required for optimizing strength properties of high yield Kraft pulps, in particular for sack paper grades.

## 2.2 High yield pulping processes

In high yield pulping, pulps that are produced have with high cellulose, lignin and hemicellulose content. This is mainly intended to preserve yield (McGovern, 1972; Christensen, 1998). During pulping, lower lignin is achieved at the expense of yield loss due to excessive dissolution of cellulose and hemicellulose. High yield pulping methods are categorized as mechanical or chemical pulping.

In mechanical pulping, fibre bundles from the wood matrix are separated by the use of mechanical energy, i.e. the energy is applied to the wood matrix. Mechanical pulps therefore bear a high proportion of short fibres, shives and fines (Helle, 1998; Jarkko, 2005). These are the unique features of mechanical pulps. Thus mechanical pulps are not suitable for those paper grades that require a high level of strength such as sack paper and linerboard. However, its pulps are suitable for paper grades such as newsprint and printing paper grades which require good formation and opacity (Helle, 1998; Jarkko, 2005). To preserve yield, yield preserving bleaching methods are used (Christensen, 1998). Pulp yield is defined as the amount of pulp produced per given wood input cooked in the digester, therefore has economical importance, e.g. the higher the yield, the more the pulp obtained from the wood purchased.

In high yield chemical pulping, pulping is done at limited hydrolysis or sulfonation (Christensen, 1998; Gullichsen *et al.*, 1999). The lignin substances in the wood matrix are only softened or partially removed due to the effect of time, temperature and cooking chemicals (McGovern, 1972; Christensen, 1998). The pulp mass is left with large amounts of

lignin. However, further lignin removal occurs during refining. Refining breaks the fibre cell walls resulting in lignin dissolution (Ebling, 1980; Helle, 1998; Anderson, 1999).

Commercially there are three high yield chemical pulping processes i.e. high yield Kraft pulping process (HYK), high yield sulphite pulping process (HYS) and neutral sulphite semi-chemical pulping process (NSSC). The HYS process produces pulp for writing and printing grades at yields of 60-90% (McGovern, 1972). Since in softwood a high content of extractives hinders sulfonation in sulphite pulping (Kocurek, 1992; Christensen, 1998), hardwoods are used as the sole raw materials.

NSSC pulps and HYK pulps have been predominately used for manufacture of packaging materials (McGovern, 1972). The NSSC process is mainly used for corrugated medium of the boxes (fluting). This process produces about 50% unfiberized mildly cooked chips, that are soft and easily defibrated compared to HYK mildly cooked chips at the same Kappa number (McGovern, 1972). NSSC pulps are produced at a yield range of 65 to 90%. Their critical quality properties are stiffness and resistance to crush (Christensen, 1998). While the main raw materials used are hardwoods, it is possible to utilize mixed pulping of hardwood and softwood in NSSC alkaline pulping (Christensen, 1998; Floris, 2000).

HYK processes have predominantly been used to produce pulps used to manufacture sack paper and Kraft linerboards in the yield range of 48-70 % (McGovern, 1972; Christensen, 1998). The critical quality properties of sack paper grade are TEA (stretch), porosity as well as bending stiffness (Pagliarini, 1992; Helle, 1998; Gurnagul *et al.*, 2005). The critical quality properties of linerboard grades are burst strength and resistance to compression, i.e. edge stiffness (Kleppe, 1985; Helle, 1998). The main raw material used for pulping HYK is softwood but hardwoods pulps are added to softwood stock furnish to improve printability (McGovern, 1972; Hell, 1998). However, it has been reported that the presence of hardwood in some paper furnish at 10-20% induces a slight reduction in tear and burst compared to pulp with 100% softwood (McGovern, 1972). Mixed pulping of softwoods and hardwoods is reported to be used occasionally in Kraft pulping processes (McGovern, 1972; Christensen, 1998). It is speculated that mixed pulping hinders proper delignification due to inherited differences in the delignification rate. As a result, for commercial application, some researchers suggest blending of pulps after pulping (McGovern, 1972; Kocurek, 1983).

Since, in high yield chemical pulping the pulp fibre bundles are left unfiberized, defibration using a disc refiner with a special plate is necessary. This is aimed only at the separation of fibre bundles, which in the mill is referred to as blowline refining (Hartler *et al.*, 1976). High

yield pulp fibres are stiff. For optimum fibre bundle separation and minimum amount of shives with minimal amounts of fines generation, hot stock defibration is adopted (Hartler *et al.*, 1976; Anderson, 1999).

## 2.3 History and development of high yield pulping

The description of the Kraft pulping process appears in several publications (Bolam, 1965; Clayton, 1969; Kleppe, 1970). The Kraft pulping process is the second generation of alkaline pulping methods. The first generation of alkaline pulping was the soda process. Soda process was predominantly used for pulping non-wood materials, mainly straw, since 1806, in England (Bolam, 1965; Kleppe, 1970).

The soda process uses sodium hydroxide as the sole cooking chemical. The first soda pulping with wood as raw material started in 1866 in Pennsylvania, USA (Kleppe, 1970; Christensen, 1998). The delignification rate in soda pulping is slow. This raised much concern about its efficiency, in particular in wood pulping. An investigation conducted by Eaton (Clayton, 1969; Kleppe, 1970), an American scientist, paved the way for successful wood pulping. He found that by using a mixture of sodium hydroxide and sodium sulphide, the delignification rate improved and strong pulps were produced. In 1870 and 1871, two US patents for the Kraft process were granted to him. This was the birth of the Kraft pulping process. Industrial application of the Kraft pulping process was made possible by Dahl in 1879 (Kleppe, 1970; Christensen, 1998). He discovered that the loss of sulphide in the chemical recovery cycle could be replaced by adding of salt cake (sodium sulphate) to the combustion furnace (Clayton, 1969). In the combustion furnace, the sulphate is reduced to sulphide. This can be re-used in the cooking chemical recovery cycle to produce white liquor. This made the Kraft process more economically viable and environmentally friendly.

Six years later after this development, the first Kraft mill was started in Sweden in 1885 (Kleppe, 1970; Christensen, 1998). Since then, due to market demand as well as due to changes in environmental regulations, various innovations have been implemented to improve the Kraft pulping technology. This includes improvements in the pulping process as well as the pulping chemistry (Christensen, 1998; Gullichsen *et al.*, 1999; Van Tran, 2005). The first generation of Kraft pulping was carried out in batch digesters. However, the drawback of the batch digester cooking system is that pulping uniformity is limited (McGovern, 1972; Harter *et al.*, 1976). The effect is reported to be more pronounced when pulping at high Kappa numbers (McGovern, 1972). Investigations on the techniques to improve pulping uniformity and optimization of pulp strength during pulping were undertaken. This included development of rapid heating displacement, e.g. super batch as replacement of conventional batch digester

cooking systems (Gullichsen *et al.*, 1999; Van Tran, 2005). This cooking principle improves pulping uniformity, resulting in less coarse rejects which improves total pulp yield (Christensen, 1998; Gullichsen *et al.*, 1999). This cooking principle involves impregnation of wood chips with high sulphide enriched liquor at the beginning of the cook (Christensen, 1998; Gullichsen *et al.*, 1999). It minimizes the effect of carbohydrate degradation due to the peeling reaction (Christensen, 1998). At the end of the cook, the concentration of caustic soda (NaOH) is kept high. This reduces the effect of lignin condensation which normally occurs as the result of lower caustic soda concentration at the end of the cook in a conventional batch digester (Kocurek, 1992; Christensen, 1998). Another pulping techniques involve the use of low temperature and keeping minimum levels of the dissolved lignin. This process is known as Lo- solid Kraft pulping (Gullichsen *et al.*, 1999; Van Tran, 2005). Figure 1 illustrates how yield and pulp strength can be improved by using different pulping techniques in a batch digester pulping system.

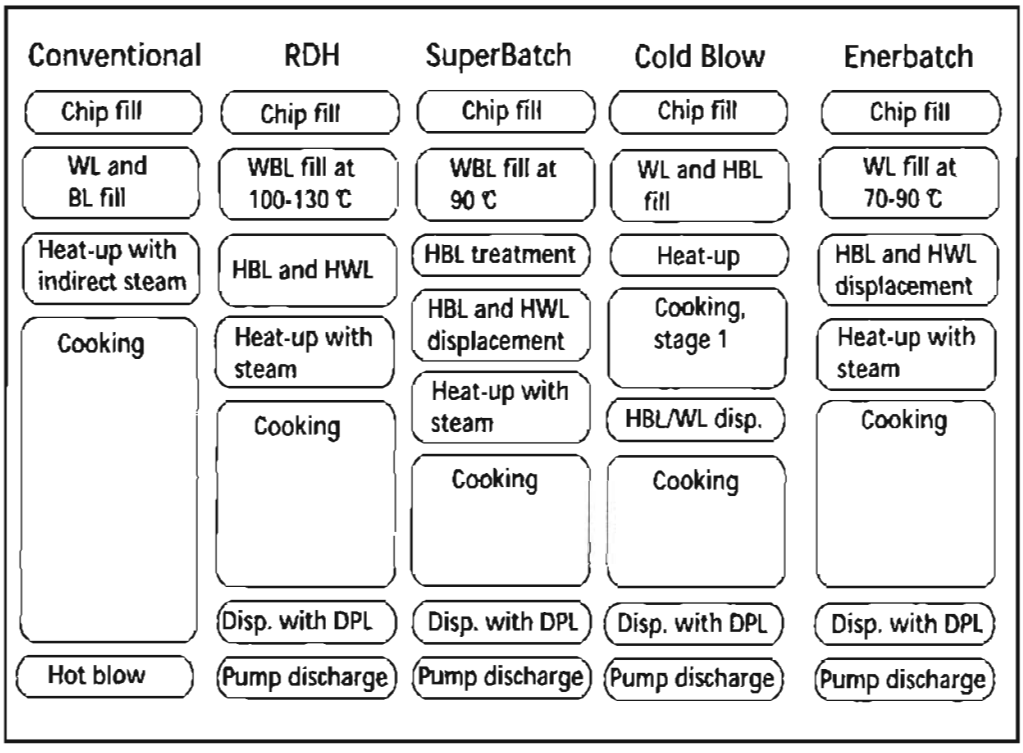



**Figure 1: Comparison between conventional and modified pulping principle in batch digesters (Gullichsen *et al.*, 1999), note RDH refer to rapid displacement heating**

The conventional continuous cooking digester was introduced as the replacement for a batch digester pulping system. It is well accepted that a continuous cooking digester has better pulping uniformity compared to a batch digester (McGovern, 1972; Gullichsen *et al.*, 1999; Van Tran, 2005). The conventional continuous cooking digester is reported to be in use since

the early 1960s (Gullichsen *et al.*, 1999). The continuous cooking digester principle allows gentle cooking through lower cooking temperatures, and during the cooking process the cooking liquor is extracted. This reduces the content of dissolved lignin resulting in the cooking chemicals reacting more mildly with cellulose (Christensen, 1998; Gullichsen *et al.*, 1999; Van Tran, 2005). Thus pulp yield is improved as well as strength properties. In the 1980's modified continuous cooking (MCC) was introduced to allow split liquor charge, which was the result of Swedish research (Christensen, 1998; Van Tran, 2005). The liquor is divided (profiled along the digester length). Figure 2 shows some important features of MCC and conventional cooking.

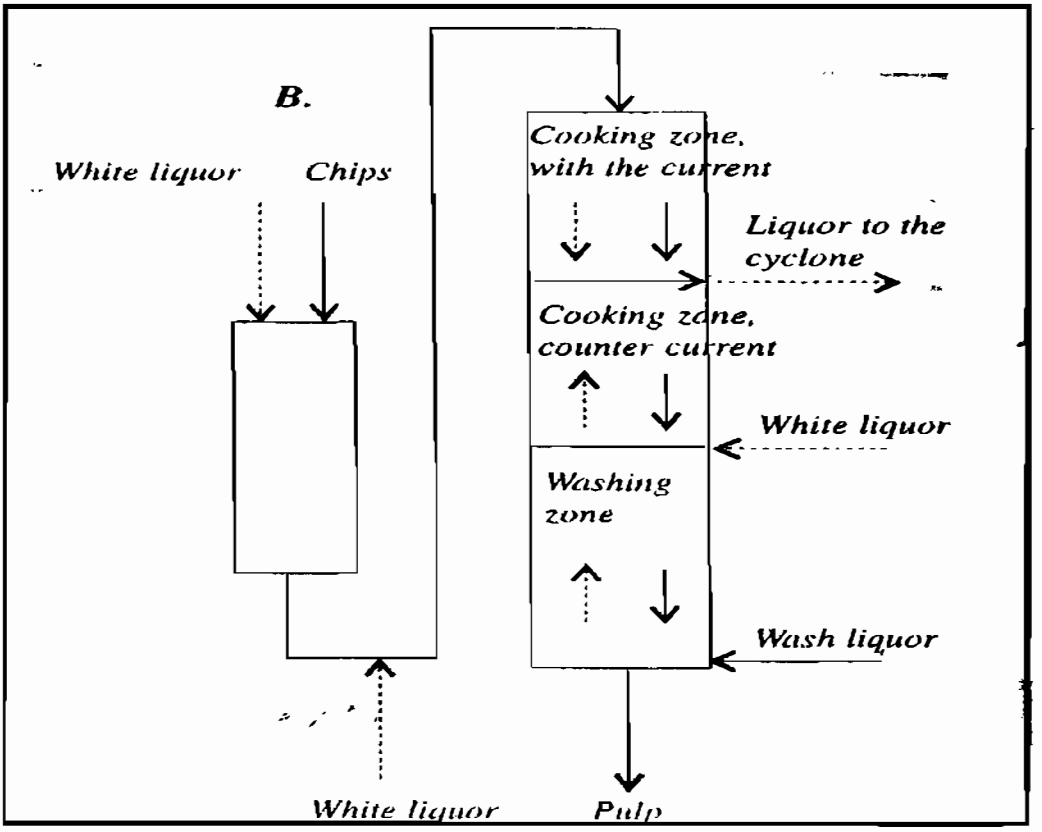



**Figure 2: Comparison between conventional (A) and modified (B) continuous pulping principle (Christensen, 1998, Gullichsen *et al.*, 1999)**

However, uncertainty surrounding the cost of raw materials and tight environmental regulations also raised the need to obtain the highest yield possible at low wood input. This enabled the use of digester additives such as anthraquinone (AQ), polysulphide (PS) and the incorporation of an oxygen delignification stage in the pulping system (Kleppe and Storebraten, 1985; Minja, 1998). Significant improvement in pulp yield as well as pulp strength properties was then recorded with an advantage of pollution load reduction (Kleppe and Storebraten, 1985; Christensen, 1998).

The modifications of the conventional processes have led to features that have been adopted in most of modern mills that exist today. The original HYK pulping process dates back to 1871 and 1882 whereby patents were published by Meech and Burn respectively (McGovern, 1972). Meech published a U.S patent on mild alkaline pulping with an in-line double cone refiner (Meech, 1871). Burns patented an alkaline pulping process with a disc refiner (Burns, 1882). This development was later modified and adopted in modern HYK pulping systems (McGovern, 1972).

It is well understood that a yield at which self-liberation of free fibres occurs, for Kraft pulping, is limited to yield of 50% (McGovern, 1972; Barker, 1996; Christensen, 1998). Thus the introduction of the disc refiner as part of a high yield pulping process enabled the defibration of mildly cooked chips, providing higher yields. Defibration allows effective washing to be done and reduces the restriction imposed by excessive coarse screening (Hartler *et al.*, 1976; Barker, 1996). As a result, a higher total yield can be achieved without the common practice of recooking coarse rejects.

HYK pulping was raised to overcome the uncertainty that was facing the American pulp and paper industry in the 1930s (McGovern, 1972). The economy was depressed, which resulted in an increase in the cost of pulp wood. As an option to reduce running costs of the paper industry, highest possible yield from the wood input was requested. The high demand of packaging materials during and after World War II also contributed to the development of the high yield pulping process (McGovern, 1972). This enabled the important development and modification of pulping and refining technology to suit these demands. Although investigations started in the 1930s, the first modern high yield Kraft mill was reported to be in operation in 1947 in the USA (McGovern, 1972). This was followed by widespread use of semi-mechanical Kraft mills in other countries such as Canada, Scandinavia, West and Eastern Europe (McGovern, 1972; Gullichsen *et al.*, 1999). This revolution also included the other two high yield chemical pulping processes: HYS and NSSC.

## 2.4 Wood source for high yield Kraft pulping

Although hardwoods delignify faster than softwoods, an important physical distinction between softwoods and hardwoods lies in the fibre length (Hegbom, 1992; Malan, 2000). Softwoods are characterized by long fibres with thin wall thickness compared to hardwood fibres (Hegbom, 1992; Malan, 2000). Fibres with thin walls have high a degree of flexibility and conformability and as a result form paper with high strength properties (Christensen,

1998). Since high yield Kraft pulps required high level of strength, the wood raw material has been restricted to softwoods (McGovern, 1972; Hegbom, 1992; Malan, 2000).

### 2.4.1 Anatomical properties of softwood

The physical features of the fibres in the wood determine the classification of the wood pulp according to end user requirements (Dinwoodie, 1965; Hegbom, 1992). However, variation exists within different species (Dinwoodie, 1965; Kleppe, 1970; Malan, 2000). These variations are accounted for in wood quality, and pulp yield as well as strength properties are affected. The important features are fibre length, cell wall thickness, coarseness, wood density, micro-fibril angle and collapsibility (Dinwoodie, 1966; Hegbom, 1992; Malan, 2000). In addition, the impact of pulping and refining processes on fibre anatomy determine the final state of the fibre (Smook, 1992; Christensen, 1998).

- **Cell wall thickness**

The collapsibility of a fibre depends on the cell wall thickness (CWT). The thinner the cell wall, the higher the collapsibility and the higher the conformability of the fibre (Christensen, 1998; Malan, 2000). In softwood, cell wall structure is based either on early or late wood. Fibres of late wood have thicker walls and fibres from early wood have thinner cell walls (Hegbom, 1992; Kocurek, 1992; Malan, 2000). The relative ratio between early wood and late wood has great influence on both pulp yield and strength properties of the softwood pulp. Early-wood produces pulp with higher strength and yield compared to latewood (Dinwoodie, 1966; Kleppe, 1970; Kocurek, 1992; Malan, 2000). The lower yield for latewood is caused by high proportion of dead cells and extractives while inferiority in strength is due to high proportion of short fibres (Kocurek, 1992) and thick cell walls so collapsibility is low.

- **Fibre length**

Softwoods are characterized by long fibres (Hegbom, 1992; Malan, 2000). The significance of the fibre length in paper making is the development of sufficient surface area for effective bonding between fibres (Helle, 1998; Malan, 2000). Below a certain fibre length limit (Helle, 1998), the surface area is small and consequently this will affect the strength of paper. However, it is very important to highlight that the influence of fibre length depends also on the pulping and refining techniques (Ebling, 1980; Hell, 1998). For instance, long fibre pulp refined at the correct refining intensity and specific refining energy will have a low fibre cutting effect and thus high strength properties (Joris, 1981; Lumiainnen, 1991).

- **Wood basic density**

Wood density is a complex wood property as it depends on a number of important characteristics such as tracheid wall thickness, diameter of tracheids, wood chemical content and the ratio of early wood to late wood. Wood density has an effect on pulp quality (Hegbom, 1992; Malan, 2000), as low wood density is characterized by lower yield as well as inferior strength properties. Lower yield is due to less number of fibres per unit volume in a wood matrix (Hegbom, 1992; Malan, 2000). While inferior in strength is contributed by the fact that low density wood constituents fibre with thick wall, fibre with thick wall has lower bonding ability (McGovern, 1972; Hegbom, 1992; Malan, 2000).

- **Reaction wood**

Due to influences of external forces (gravity on branches or prevailing winds), compression wood is produced (Hegbom, 1992; Kocurek, 1992). Compression wood is characterized by wider annual rings with shorter fibres (Hegbom, 1992). It has more lignin and hemicellulose than normal wood. Compression woods do not respond well to pulping and are a source of knots (Hegbom, 1992; Kocurek, 1992). Knots are the source of high coarse screen rejects, thus affecting pulp yield.

### 2.4.2 Chemical composition of softwood

Wood chemical constituents are important in pulp and paper making (Kocurek, 1992; Christensen, 1998), and largely determine the economic benefit of the wood. For instance wood with a high lignin or extractives content consumes more cooking chemicals and may require a longer cooking time to achieve the same Kappa number as compared to wood with a lower lignin or extractives content (Kocurek, 1992; Smook, 1992). This reduces productivity and increases costs of cooking chemicals. Wood with a high content of hemicellulose and cellulose gives a high yield with improved pulp quality (Kocurek, 1992; Christensen, 1998). This maximizes the utilization of wood in fibre processing, resulting in higher yield per unit of wood input.

It is well accepted that optimisation of wood fibre processing must be complemented by the improvement of the chemical components as well as physical wood components of the trees. This has been demonstrated in the northern hemisphere countries (Per-Michael, 2002, Venter, 2005), trees are genenetically modified to meet particular requirements of paper or to improve pulp yield. Figure 3 shows wood fibre wall structures of softwood which are made of various layers (P=primary cell wall, $S_1$, $S_2$ and $S_3$ = outer, middle and inner portions of secondary cell wall respectively while M is the middle lamella).

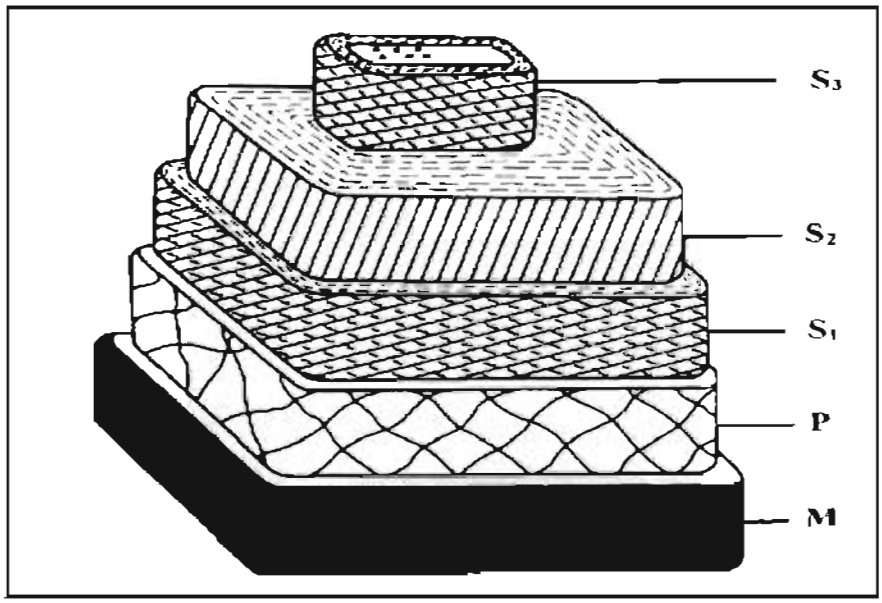



**Figure 3: Main layers of a typical softwood fibre structure (Gullichsen *et al.*, 1999)**

- **Cellulose**

The majority of the cellulose is found in the middle and inner portion of the secondary wall layers ($S_2$ and $S_3$) as shown in Figure 3. Cellulose has been described as a linear high polymer of d-glucose unit with a crystalline structure (Christensen, 1998; Gullichsen *et al.*, 1999). As a result it is not easily accessible to pulping chemicals and is difficult to dissolve in water and other organic solvents (Christensen, 1998). Its degree of polymerisation (DP) is between 10000-20000 units depending on the species (Christensen, 1998; Gullichsen *et al.*, 1999). Figure 4 shows the cellulose structure of softwoods.

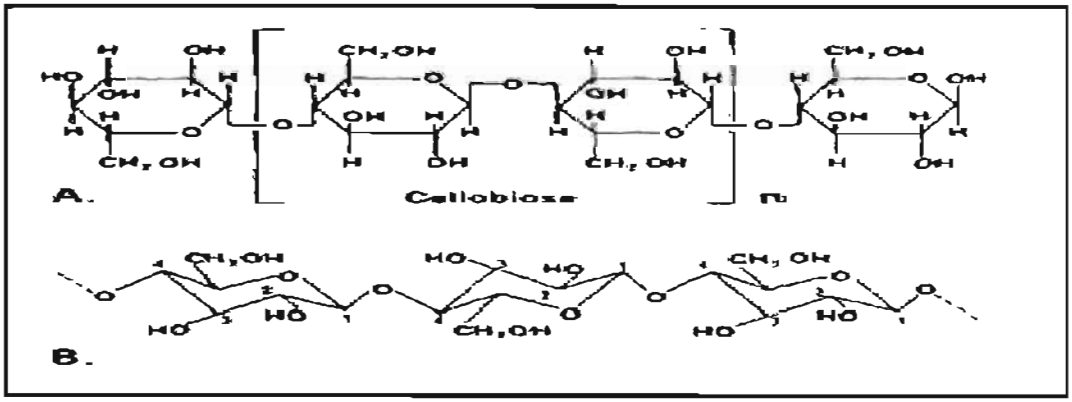



**Figure 4: Typical softwood cellulose structure (Christensen, 1998; Gullichsen *et al.*, 1999)**

- **Hemicelluloses**

The middle lamella structure M and $S_2$ layer are rich in hemicelluloses (Figure 3). They is a group of branched polysaccharide polymers built up with glucomannan, xylan and galactan (Smook, 1992; Christensen, 1998). The percentage of hemicelluloses in softwood is in the range of 14-18%. Hemicellulose acts as a binding agent between the crystalline cellulose microfibrils. It swells very easily in water (Christensen, 1998). This is an important feature to the paper maker as it is the basis for the formation of interfibre hydrogen bonds (Smook, 1992). However, due to its amorphous structure it is easily attacked by cooking chemicals and, as a result, during chemical pulping it easily dissolved (Christensen, 1998). However, in high yield Kraft pulping appreciable amounts of hemicellulose are retained (McGovern, 1972). The effect on wood chemical components during high yield Kraft pulping discussed in detail in section 2.5.1.

- **Lignin**

The lignin is located mainly in the middle lamella (m) and in the primary cell wall (p) (Stephen, 1992; Christensen, 1998). Its content in softwood is reported to be about 28% (Smook, 1992; Stephen, 1992). Lignin is a thermoplastic polymeric compound with a softening temperature of about 140-180 °C (Kocurek, 1992; Christensen, 1998). The important step in Kraft pulping is delignification by alkaline hydrolysis of phenolic ether bonds, whereby lignin becomes soluble in alkali (Smook, 1992; Christensen, 1998). From a high yield pulping perspective, the lignin is only softened without substantial removal. As a result, high yield pulp contains a high content of lignin, and thus its fibres are stiff (McGovern, 1972; Hartler *et al.*, 1976). Figure 5 shows a typical lignin structure found in softwood.

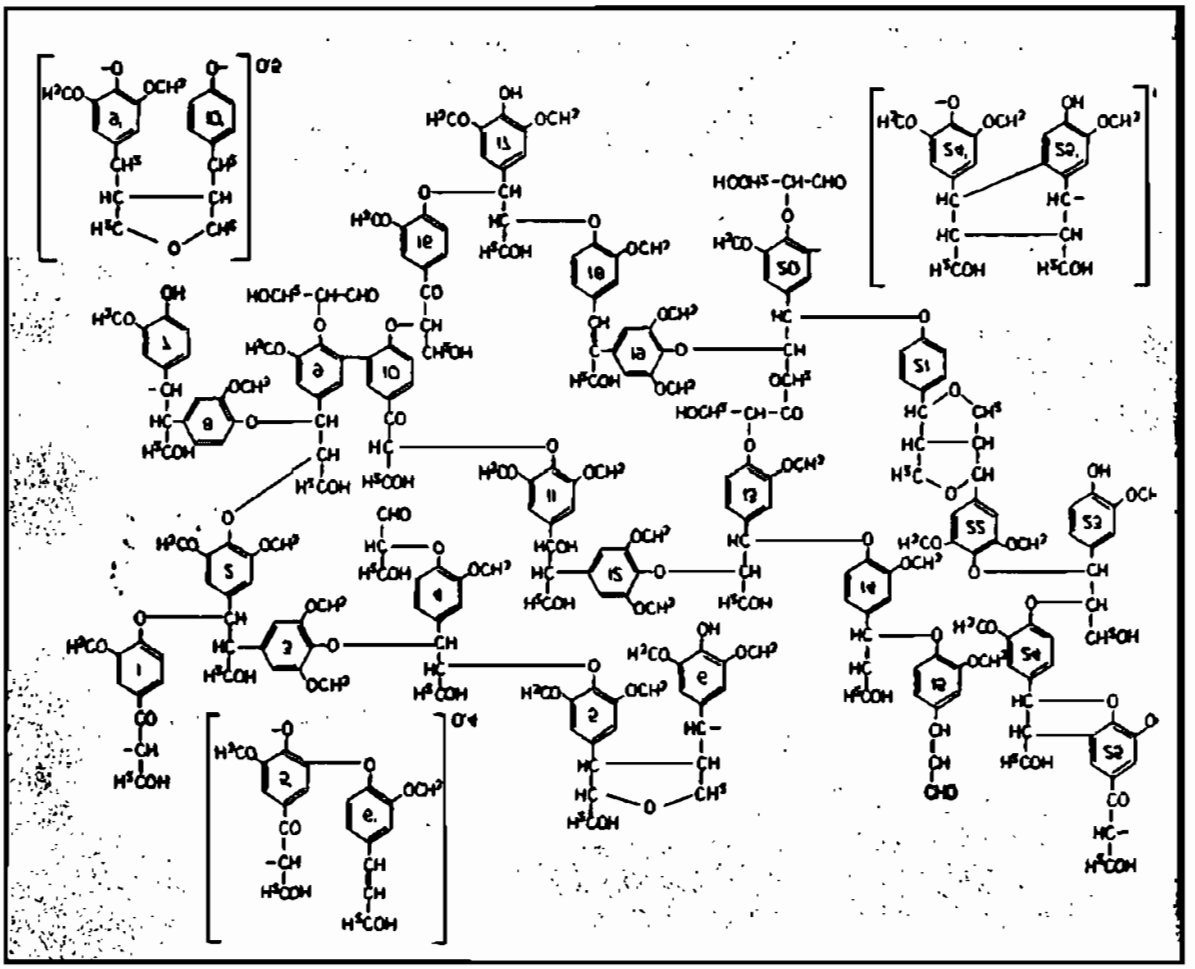


**Figure 5: Typical of lignin structure found in softwoods (Smook, 1992; Stephen and Carlton, 1992; Christensen, 1998)**

- **Extractives**

Generally softwoods contain high amounts of extractives in the range of four to five percent compared to hardwoods which are in the range of two to three percent (Smook, 1992; Kocurek, 1992; Gullichsen *et al.,* 1999). Extractives are found in the parenchyma cells (Christensen, 1998). Softwoods contain an additional canal system which contains resin acids and esters dissolved in turpentine (Gullichsen *et al.*, 1999). The effects of extractives during Kraft pulping have been investigated (Kocurek, 1992; Smook, 1992; Christensen, 1998; Gullichsen *et al.*, 1999). The main claim from these authors is that extractives cause higher consumption of cooking chemicals, pitch formation and foaming problems. It has been shown that phenols prevent lignin from becoming sufficiently hydrophilic and increases the chances of condensation (Kocurek, 1992; Christensen, 1998). Phenyl-propane units, which are derived from coniferyl alcohols display lignin-like reactions during Kraft pulping (Kocurek, 1992; Christensen, 1998).

The effects of extractives in Kraft pulping can be minimized in a number of ways, which include the use of alkaline treatment of the wood chips which helps to saponify the ester bonds and neutralize the free acids (Smook, 1992; Christensen, 1998). The soap formed (R-COONa) helps to disperse the unsaponifiable fraction of unreacted extractives (Christensen, 1998). Another option is the ageing of logs or chips to hydrolyse fatty acid and enzymatically or oxidatively break down the double bonds (Christensen, 1998). The other method is pre–steaming of the wood chips to drive off volatile compounds before digestion (Smook, 1992; Christensen, 1998). Figure 6 shows typical extractives found in softwoods.

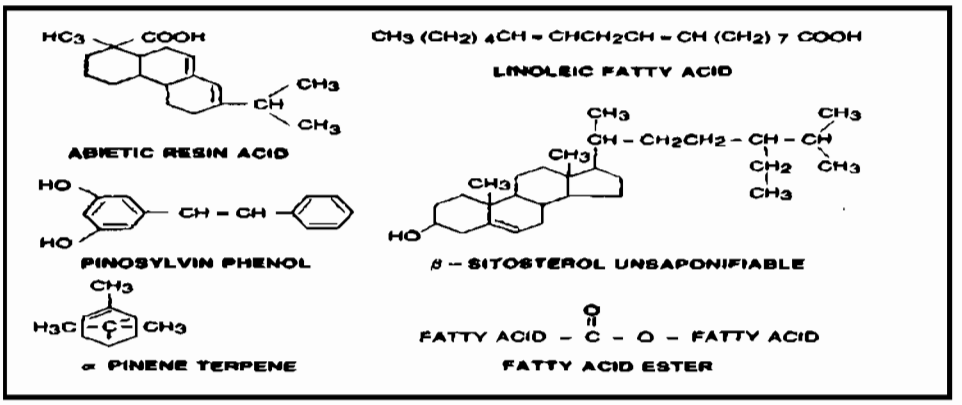



**Figure 6: Typical extractives found in softwoods (Smook, 1992; Christensen, 1998)**

## 2.5 Kraft pulping reactions

### 2.5.1 Pulping reactions

The Kraft cook is a typical liquid –solid reaction system (Kocurek, 1992; Gullichsen *et al.*, 1999), using strong alkaline liquor with a pH value of approximately 14. For pulping applications (cooking liquor) $Na_2S$ and NaOH are expressed as active alkali (AA) based on [$Na_2S$] and [NaOH] or effective alkali (EA) based on 1/2[$Na_2S$] and [NaOH]. Hydrolysis of the cooking chemicals during Kraft pulping results in $OH^-$ and $HS^-$ ions in the pulping solutions (Kocurek 1992; Christensen, 1998):

$$Na_2O + H_2O ==== 2NaOH$$

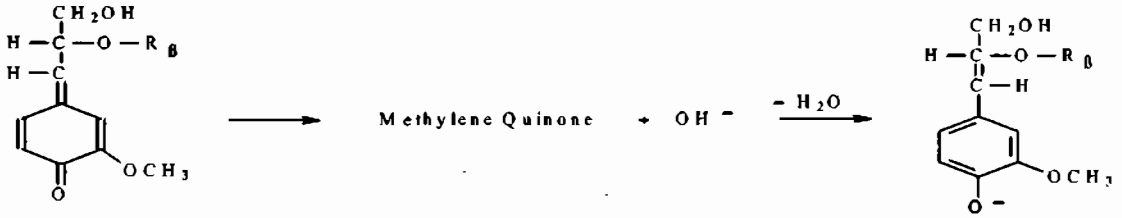
$$Na_2S + H_2O == NaOH + NaSH$$

Kraft pulping involves the hydrolysis of both glucosidic bonds for cellulose and a- ß alkyl ethers, alkyl-aryl ethers and methoxy groups for lignin (Stephen and Carlton, 1992; Christensen, 1998; Gullichsen *et al.*, 1999). The hydrolysis of glucosidic bonds is very noticeable at temperatures above 100 °C (Kocurek, 1992; Christensen, 1998). Most studies have shown that although some side reactions occur during Kraft pulping (Kocurek 1992;

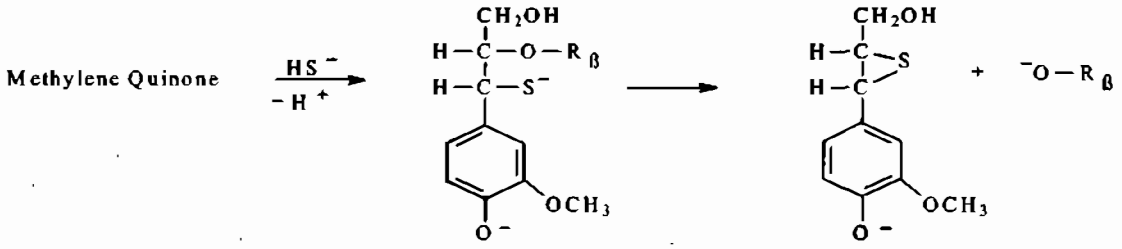
Smook, 1992; Christensen, 1998), the one of concern for the Kraft pulping industry is the alkaline peeling reaction which is associated with degradation of cellulose. The peeling reaction may lead to excessive cleavage of cellulose which affects the degree of polymerization (DP) and consequently lowers pulp yield and produces pulp with inferior strength properties (Kocurek, 1992; Christensen, 1998). In an attempt to reduce the effect of the peeling reaction and thus improve pulp quality, various pulping technologies have been developed (Christensen, 1998; Kubes *et al.*, 2002; Van Tran, 2005), as discussed in detail in section 2.2.3 and 2.2.5. These modified Kraft pulping methods make the delignification reaction more selective (Christensen, 1998; Van Tran, 2005).

In HYK pulping, pulping is done at a limited hydrolysis level and thus a large amount of cellulose and hemicellulose are left in the pulp. Hydrolysis of a- ß alkyl ethers and alkyl-aryl ethers bonds of lignin occurs at temperature range of 140-180 °C (Kocurek, 1992; Christensen, 1998). In HYK pulping, pulping is done at a limited delignification level. This involves either softening or partial removal of the lignin due to the effect of temperature and cooking chemicals (McGovern, 1972). The temperature softens the lignin in the wood matrix and hydrolysis breaks or weakens the a- ß alkyl ether and alkyl-aryl ether bonds. Thus HYK pulp contains large amount of lignin. However, further lignin dissolution occurs during refining (Ebling, 1980; Hell, 1998). Refining breaks the fibre cell walls and the lignin fragments are removed from the fibre through leaching (Lindstrom *et al.*, 1978; Li and Macleod, 1992). Mechanisms for the lignin reaction during Kraft pulping have been reported in several studies (Stephen and Carlton, 1992; Kocurek, 1992; Christensen, 1998) and can be outlined as follows:

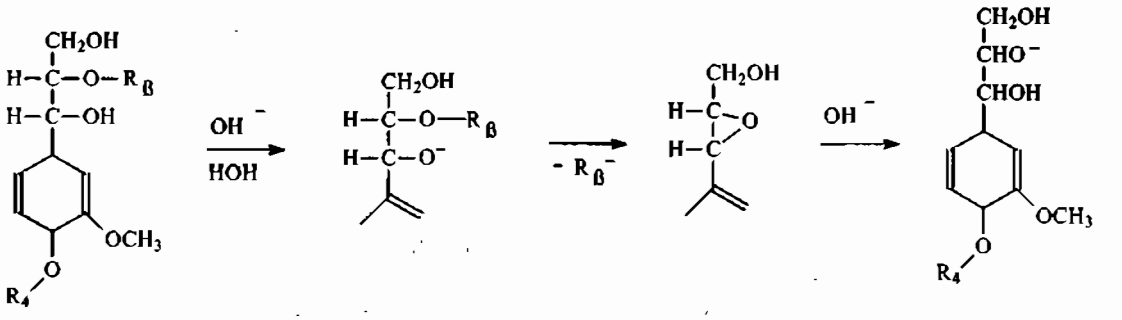
1) The breakdown of the ether bonds which comprises about 75% of inter-unit bonds (Christensen, 1998). Figure 7a-d depicts the typical reaction mechanisms during Kraft pulping. Initially, attack occurs on the hydroxyl groups, and the phenoxide ion formed eliminates an alkyl group from the a-carbon atom as the reaction shown in (Figure 7a).
2) This is followed by the nucleophilic attack by an $OH^-$ and $HS^-$ ion, formation of an epoxide ring and the cleavage of the ß- aryl ether from methylene quinone structure due to reaction with $HS^-$ which is a very strong nucleophilic reagent (Stephen, 1992) as shown in Figure 7b.
3) Further reaction of a a-sulphonic acid gives ß-elimination in alkali (Christensen, 1998) as shown in figure 7c. Because the methoxy groups (R=H or $CH_3$) are unstable towards both $OH^-$ and $RS^-$, they under go demethylation leading to the formation of sulphur containing compounds: either $CH_3SH$ (methyl mercaptane), $CH_3SCH_3$ (dimethyl sulphide) or $CH_3SSCH_3$ (dimethlydisulphide) which cause the rotten egg odour at Kraft mills. The formation reaction is shown in Figure 7d.



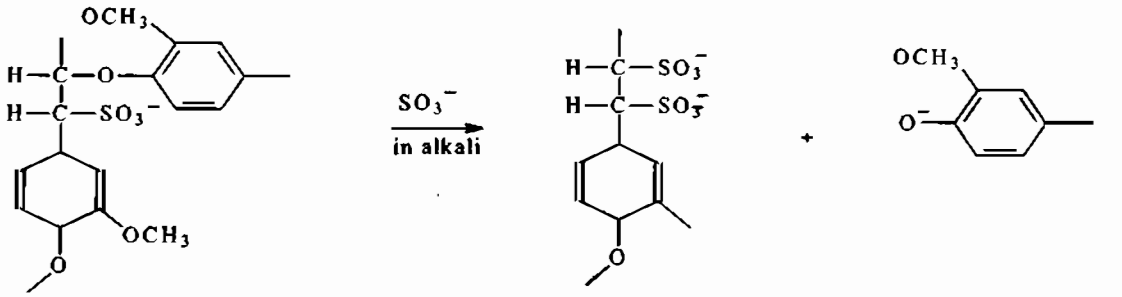

(a)



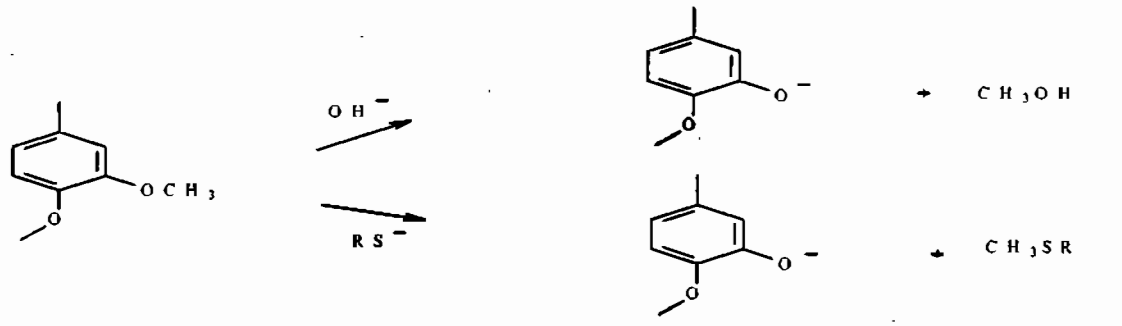

(b)




(c)




(d)




(e)

**Figure 7a-e: Softwood lignin reactions during Kraft pulping (Stephen and Carlton, 1992; Kocurek, 1992; Christensen, 1998)**

4) The condensation reactions. A high concentration of many types of dissolved acids and lignin-Na salts may lead to a low pH towards the end of the cook. The effect is more pronounced when the pH falls below 10.5 (Christensen, 1998). This causes dissolved lignin to re-precipitate (condense) on fibres due to lower residual alkali (Christensen, 1998). This happens particularly in conventional Kraft pulping which is associated with a low concentration of NaOH at the end of the cook (Christensen, 1998; Van Tran, 2005). Condensed lignin is reported to be more difficult to remove (Kleppe, 1970; Christensen, 1998). Figure 8 shows reaction mechanisms which lead to condensation reaction.

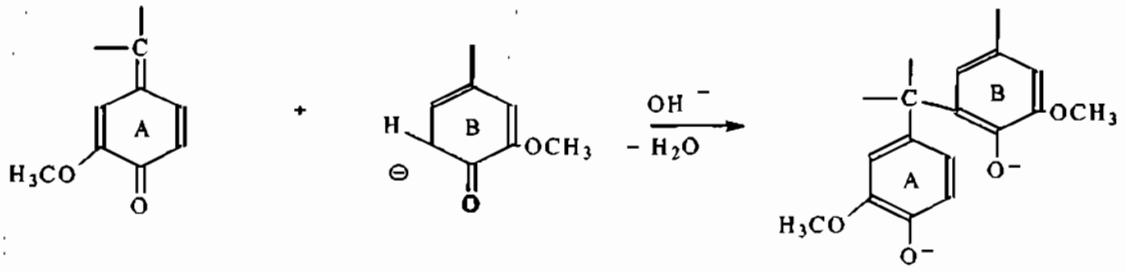



**Figure 8: Typical lignin condensation reactions in alkaline liquor (Stephen, 1992; Christensen, 1998)**

The use of liquor profiling and rapid heat displacement pulping techniques reduces the risk of lignin re-precipitation in Kraft pulping (Christensen, 1998; Van Tran, 2005). These pulping techniques ensure a high level of cooking chemicals (as NaOH) at the end of the cook. This prevents the precipitation of lignin back on to the fibre (Gullichsen *et al.*, 1999). For process monitoring purpose, both the black liquor pH and residual alkali is reported. This helps to evaluate how well the cook has been performed. For good pulping practice (Kocurek, 1992), the residual alkali level is reported to be in the range of six to seven gram per litre for a pulp yield of less than 70% and seven to nine gram per litre for a pulp yield of above 70%.

### 2.5.2 Delignification kinetics during Kraft pulping

Delignification in Kraft pulping occurs in three distinct phases (Kleppe, 1970; Christensen, 1998; Kubes *et al.*, 2002). In the early phase only a small amount of lignin is dissolved which involves cleavage of phenolic ß-aryl ethers (Kocurek, 1992; Christensen, 1998). This depends on the presence of $HS^-$ while $OH^-$ is required for the ionising the phenolic group, the diffusion out of fibre being the rate -controlling step with an activation energy as low as 40 kJ/mol (Kocurek, 1992).

When the temperature reaches about 140 °C this phase is practically over (Kocurek, 1992; Christensen, 1998). The delignification rate in this phase is a zero order reaction with respect to the lignin removal (Kocurek, 1992; Christensen, 1998). The removal of the bulk of the lignin proceeds only at a temperature above 150 °C (Kleppe, 1970; Kocurek, 1992), where the alkali consumption is fairly low and is associated with the cleavage of non-phenolic ß-aryl ether links (Christensen, 1998). Further lignin breakdown depends on the cleavage of the non-phenolic ß-ether. The lignin dissolution depends highly on the presence of $OH^-$ ion concentration rather than $HS^-$ ion (Kocurek, 1992; Christensen, 1998). The delignification rate in this phase is more controlled by the chemical reaction. The reaction is of first order reaction with respect to the lignin removal with activation energy of 120-150 kJ/mole (Christensen, 1998; Kubes *et al.*, 2002).

The residual delignification phase is reported to be slow (Kleppe, 1970; Christensen, 1998; Kubes *et al.*, 2002). It is speculated that this is due to a low lignin content remaining in the wood. However, other studies have reported that condensed fragments of lignin are responsible for the slow rate (Kleppe, 1970; Christensen, 1998).

Many kinetic models been developed to describe the phases of Kraft pulping (Kerr, 1970). The kinetics of Kraft pulping seem to be complicated. No formula has yet been published to describe the rate of delignification for the entire Kraft pulping process. This may be due to difficulties in monitoring the changes of $[OH^-]$ and $[SH^-]$ during industrial pulping. On other hand, the rates of lignin removal differ from one phase to the other, thus making it more difficult to develop general kinetics equations to describe the entire Kraft phase. It is generally accepted that for process control purposes, the H-factor remains the best option.

### 2.5.3 Influence of pulping variables on pulp quality

- **Wood chips size**

The effects of size of wood chips on the overall pulping results are already reported in many studies (Kleppe, 1970; Bublitz and McMahon, 1976; Kocurek, 1992). It is well established that the diffusion of hydroxyl ions to the reaction sites depends on the wood chip thickness. Thus if the chip is too thick, the delignification may proceed much faster on the surface than towards the centre of the chip (Smook, 1992; Kocurek, 1992; Christensen, 1998) because the penetration of the cooking chemicals is impaired. This results in non-uniform pulping and consequently affects pulp quality and yield.

In 1976, Bublitz and McMahon studied the influence of chip size reduction by shredding. They found that shredded chips improve pulp yield. This finding was earlier reported in 1970 by Kleppe. In this study, it was found that chip shredding resulted in a yield increase of 0.5-1% at the same Kappa number compared to convectional chip size. This is because with reduced woodchip size, uniformity of delignification is enhanced. However, the shortcoming of the shredded chips is that they produce pulp with inferior tear strength (McGovern, 1972).

This problem is more pronounced in conventional pulps compared to high yield pulps (Kleppe, 1970; McGovern, 1972). Thus some publications have insisted on the need to use wood chips with a small size for high yield Kraft pulping (McGovern, 1972). This is because high yield pulps are cooked for a short time with a low liquor charge compared to conventional pulps. Thus effective impregnation which results in uniform pulping can be achieved with small wood chip size compared to conventional pulping, and this is very important particularly in batch digesters. Variations in woodchip size affects pulp yield due to high screen rejects as well as results to Kappa number variation which affects pulp strength properties.

- **Cooking temperature**

The cooking temperature provides the driving force for the pulping reactions, and thus controls the pulping rate (Smook, 1992; Kocurek, 1993; Christensen, 1998). An increase of 10 °C doubles the reaction rate (Vroom, 1957). The temperature used is normally in the range of 150-175 °C for batch digesters and 150-170 °C for continuous digesters (Christensen, 1998; Gullichsen *et al*, 1999). However, under modified Kraft pulping i.e. Lo-solid Kraft pulping, it is possible to cook at lower temperature as low as 143-145 °C (Gullichsen *et al.*, 1999; Van Tran, 2005).

Delignification rate in Kraft pulping is quantitatively expressed according to the Arrhenius equation $K_r=Ae^{-Ea/RT}$ (Vroom, 1957; Kocurek, 1992). This equation is used to evaluate the reaction rate constant at any temperature (Appendix A-1). The relative reaction rate corresponding to the temperature at particular time is plotted vs. the cooking time; the area under the curves gives a single numerical value called the H-factor. The H-factor has been successfully employed in the digester operation to predict the yield as well as target Kappa number (Christensen, 1998; Kocurek, 1992).

It is well understood that pulp yield depends on both delignification and the degree of carbohydrate degradation. The degree of carbohydrate degradation is determined by the pulp viscosity (Kocurek, 1992; Christensen, 1998). Therefore, the shortcoming of the H-factor is

that it cannot be used to predict pulp viscosity (Kocurek, 1992). This is because the activation energy for cellulose chain cleavage is not the same as that of delignification (Kocurek, 1992; Christensen, 1998). In addition, it has been demonstrated in earlier studies, that cellulose cleavage has higher activation energy (179 kJ/mol) compared to the delignification reaction (134 kJ/mol) (Kocurek, 1992; Kubes *et al.*, 2002). Thus pulping to higher temperature has more effect on cellulose than on lignin removal. Due to the higher activation energy of cellulose, pulping at higher temperature results in a faster drop in viscosity due to severe cleavage of cellulose. It is generally accepted that although pulping at higher temperature can shorten cooking time, resulting in improved digester productivity, severe attack on cellulose jeopardises the advantages. This phenomenon explains why temperatures above 180 °C are seldom used.

- **Cooking liquor charge**

The degree of delignification, among other factors, is influenced by the liquor charge (Kocurek, 1992; Smook, 1992). Generally, the liquor charge (expressed as $Na_2O$ or NaOH, on oven dry mass of wood chips needed) ranges from 18-24% for the lower yield and 10-16% for high yield pulp (Christensen, 1998; Gullichsen *et al.*, 1999). Sufficient liquor charge is required to ensure the delignification reaction takes place effectively and some liquor is left to neutralize acid salts formed during pulping (Kocurek, 1992; Christensen, 1998).

The influence of liquor charge on pulp yield has been investigated (Kleppe, 1970; Kocurek, 1992; Smook, 1992; Christensen, 1998). It is reported that an increase of 1% effective alkali (EA) based on wood, has no significant impact on total yield of softwood. It is speculated that at a very small liquor charge only a decrease in retention of xylan, with little impact on retention of glucomannans occurs (Kleppe, 1970). However, at an increased liquor charge the negative effect on pulp quality is obvious due to excessive dissolution of carbohydrates (Kocurek, 1992; Smook, 1992, Christensen, 1998).

- **Liquor to wood ratio**

The liquor to wood ratio is the proportion of total liquid in the digester to oven dry mass of chips charged. Its implication is that all the chips in the digester must be saturated by the liquor/ water solution to allow effective liquor impregnation of the wood chips in the entire digester (Smook, 1992; Christensen, 1998).

After the normal charge of liquor, an extra amount of black liquor is added to make up the liquor to wood ratio required (Kocurek, 1992; Smook, 1992). Black liquor is preferred to pure water because it increases the sulphidity which enhances delignification selectivity (Christensen, 1998). It also enhances the concentration of dissolved solids in the liquor and

thus reduces steam demand to evaporators, resulting in an energy saving (Kocurek, 1992). However, the presence of dissolved lignin in black liquor has reported to inhibit pulping to lower Kappa numbers (Kocurek, 1992).

To ensure that all the wood chips are saturated with liquor in the digester, a liquor to wood ratio in the range of 3-5:1 is selected (Kocurek 1992; Christensen, 1998). Effective liquor impregnation of wood chips prior to cooking enhances uniform pulping (Kocurek, 1992; Smook, 1992). This is more important in HYK pulping because this is carried out at a short cooking time. The use of nitrogen gas at two bars to pressurize conventional batch digesters prior to cooking is adopted (Christensen, 1998). In a flow- through digester, the digester is pressurized by means of circulating liquor by pumps, before cooking (Kocurek, 1992). These two options are described as the best ways to achieve effective liquor impregnation of wood chips and hence uniform pulping in batch digesters (Kocurek, 1992).

- **Sulphidity of the cooking liquor**

The Kraft cooking liquor is a solution of NaOH and $Na_2S$ in water. The percentage of $Na_2S$ in relation to the entire amount of active alkali or effective alkali is referred to as sulphidity (Smook, 1992). Its role during cooking is speculated to be an increase of cooking rate and retention of carbohydrates by reducing the effect of alkaline peeling (Kocurek, 1992; Christensen, 1998).

The effect of liquor sulphidity for various wood species has been demonstrated in several studies (Smook, 1992; Kocurek, 1992; Christensen, 1998; Gullichsen *et al.*, 1999). Most of these authors have claimed that liquor with higher sulphidity improves yield and pulp strength. In all these investigations, their results showed that when sulphidity falls below 15% a significant negative effect on pulp quality occurs. Thus it is advised that the sulphidity must be kept higher (20-35%). High sulphidity, as high as 40%, may give good results. However, accelerates corrosion in the digester (Kleppe, 1970; Kocurek, 1992), and thus for commercial process it is avoided.

## 2.6 Kraft pulping techniques for improving pulp yield

### 2.6.1 Pulping to higher Kappa number

It is well accepted that the higher the Kappa number, the higher the yield, thus the better the economic benefit released from the wood resource. However, although there are technical and economic reasons which support the need to explore further the potential of high yield

Kraft pulps compared to the Kappa range currently applied in industry, little information on high yield Kraft pulps has been reported. In this study it was investigated in detail.

### 2.6.2 Polysulphide Kraft pulping

The use of polysulphide (PS) as an additive to Kraft liquor was first patented by Fuller and Woodside (Kleppe, 1970; Christensen, 1998) in the USA in 1943. In an attempt to understand the kinetics of polysulphide (Kraft-PS), several studies have been conducted (Christensen, 1998., Gullichsen *et al.*, 1999; Kubes *et al.*, 2002). The main claim from those researchers is that the effect on carbohydrate degradation due to the peeling reaction is reduced, resulting to improved pulp quality and yield.

It is well understood that sodium polysulphide ($Na_2S$ +S: $Na_2S_n$) oxidizes oxycellulose to –COOH ($C^1$ and $C^6$). Oxidized oxycelluloses are stable in alkali, and are less degraded during pulping (Christensen, 1998; Kubes *et al.*, 2002). Carbohydrate analysis of polysuphide pulps has shown that the yield increase is due to retention of glucomannan in softwoods due to reduced peeling reaction (Kleppe, 1970; Kubes *et al.*, 2002). Studies on the delignification rate have shown that Kraft-PS is faster than in a conventional Kraft cook (Kleppe, 1970; Kubes *et al.*, 2002). This gives an advantage of cooking for a shorter time (lower H-factor) resulting in improved digester productivity.

At Peterson mill in Norway, the use of PS as digester additives has been reported since 1973 (Christensen, 1998). In 1972, McGovern reviewed progress in HYK pulping in Northern America and Europe and reported that one mill in Poland, by using PS in pulping spruce, improved yield by 10-15% from a normal yield of 50%. Good results in terms of pulp quality and delignification rate improvement (Kleppe and Storebraten, 1985; Kubes *et al.*, 2002) are reported with the addition of anthraquinone (Kraft-PSAQ). AQ accelerates delignification and stabilizes carbohydrates against the peeling reaction (Kleppe and Storebraten, 1985; Minja, 1998; Kubes *et al.*, 2002). These effects of AQ in Kraft pulping are applicable to both softwood and hardwood species (Kubes *et al.*, 2002). The impregnation of wood chips with sulphide enriched liquor (black liquor, green liquor or hydrogen sulphide) prior to pulping has also been shown to improve pulp quality (Olm, 1996; Bykova *et al.*, 1997).

### 2.6.3 Delignification of high yield pulps by oxygen and alkaline conditions

Extended delignification involves two stages of delignification (Kleppe and Storebraten, 1985). The first stage involves pulping to higher Kappa number, e.g. 90-110 either by

conventional or modified pulping methods. The pulps are then further delignified by the use of oxygen and alkali (Kleppe and Storebraten, 1985). The Kappa number is reduced to about 30-50% without a negative effect on neither total yield nor strength properties (Kleppe and Storebraten, 1978; Kleppe and Storebraten, 1985; Minja, 1998).

This process was reported to be developed in the 1960s, however, industrial application was reported in 1975 (Kleppe and Storebraten,, 1985; Minja, 1998). The unique characteristics of oxygen delignified high yield pulps or polysulphide pulps is that they require less refining energy when compared to pulp produced by conventional methods at the same Kappa number (Kleppe and Storebraten,, 1985; Minja, 1998). This gives an advantage of minimizing production costs related to the refining process. This has been described as one of the reasons for their dominance in producing high yield Kraft pulps e.g. Sappi Tugela mill (McGovern, 1972; Hartler *et al.*, 1976; Kleppe and Storebraten, 1985).

Perhaps the best example to explain this is the study conducted by Kleppe and Storebraten (1985), in which they showed that either polysulphide or conventional Kraft pulping of Norwegian spruce to Kappa number 100-110 followed by alkaline oxygen delignification produced pulp with less shives and required less refining energy at the same Kappa number compared to conventional Kraft pulp. These pulps did meet the quality specification of sack paper. It was also demonstrated that polysulphide Kraft pulping to Kappa number 110-150 followed by alkaline oxygen delignification produced a pulp which meets the specification for linerboard.

### 2.6.4 Alkaline peroxide treatment of high yield pulps

Alkaline peroxide mechanical pulping (APMP) is one of the most recent special processes developed (Jarkko, 2005). This process involves the modification of lignin present in high yield pulps. It is well understood that in order to develop high strength from high yield pulps and minimize refining energy, some kind of lignin modification during refining of high yield pulps is required. This includes alkaline peroxide treatment of the high yield pulps prior to HC refining (Jarkko, 2005). Alkaline peroxide oxidizes lignin to carboxylate groups. It has been shown that carboxylate groups participate in the hydrogen bonding (Jarkko, 2005), thus improving the fibre bonding ability. There are no losses in yield, as lignin is only modified rather than being removed.

The best example of studies to describe modification of lignin present in high yield pulps is by the forest research in India (FRI (http://www.corgfre./institutes2/research). In this study they found a significant energy reduction during refining of modified pulp (350 kwh/t from 500 kwh/t). In addition, the results in these studies indicated that strength properties can be improved: tensile index by 30-140%, burst index by 18-140% and tear index by 14-150%. This would then automatically enhance the productivity for the same input of woods, with the advantage of saving energy and pulping chemicals. More importantly, this method allows production of pulps for almost all applications (Jarkko, 2005). However, these findings were based on hardwood (Birch). It will be interesting and important to find out how high yield softwood pulps respond to this type of treatment, which was not part of this study.

## 2.7 Characteristics of high yield Kraft pulps

### 2.7.1 Effect of Kappa number on pulp strength properties

The effect of Kappa number increase on pulp strength has been reported (Jones, 1972; McGovern, 1972; Hartler *et al.*, 1976). These effects can be summarized as follows:

- The pulp tends to be slow in drainage, and consequently limits the paper machine speed and accelerates web breakage (Helle, 1998). This is due to a high amount of fines formed during refining as the result of brittleness and stiffness of fibres (Hartler *et al.*, 1976).
- A dark pulp colour is produced at higher yield. This due to the high content of lignin.
- High content of shives. It is generally accepted that pulp at Kappa number above 90 will contain high content of shives (Kurdin, 1981). This is due to limited pulping uniformity.

### 2.7.2 Bleachability of high yield Kraft pulp

In 1972, McGovern, reported the effect of bleaching of high yield pulps. Bleaching affects pulp yield significantly. In one study it was found that unbleached yield of high yield Kraft pulp of 62% was reduced to 50% (McGovern, 1972). This obviously is considered as uneconomical. Thus the advantage of the high yield can be preserved by the use of the lignin preserving methods (Christensen, 1998; Jarkko, 2005). The use of an oxygen delignification stage has also been described as improving bleachiability (McGovern, 1972; Kleppe and Storebraten, 1985). High yield pulps are always left with large amount of shives due to pulping non-uniformity (Christensen, 1998). According to Kleppe and Storebraten (1985), oxygen delignification also reduces shives. This is important because shives consume more bleaching chemicals (McGovern, 1972; Kleppe and Storebraten, 1985; Christensen, 1998).

## 2.8 Refining

### 2.8.1 High consistency refining process

HC refining refers to a refining operation which is carried out in an atmospheric discharge refiner. The pulps are fed in the refiner at about 28-40% dry solids content using a screw feeder. HC refining was introduced in the 1960s as part of refining techniques to improve stretch as well as TEA (Pagliarini, 1992). This is beneficial to extensible paper grades such as sack paper which require a high level of TEA e.g. in cement packaging (Pagliarini, 1992; Helle, 1998; Gurnagul *et al.*, 2005). In addition to that, to enhance TEA as well as stretch, the sheet is allowed to shrink freely under little tension during drying (dryer cylinders operated at a loose draw) as illustrated in Figure 9.

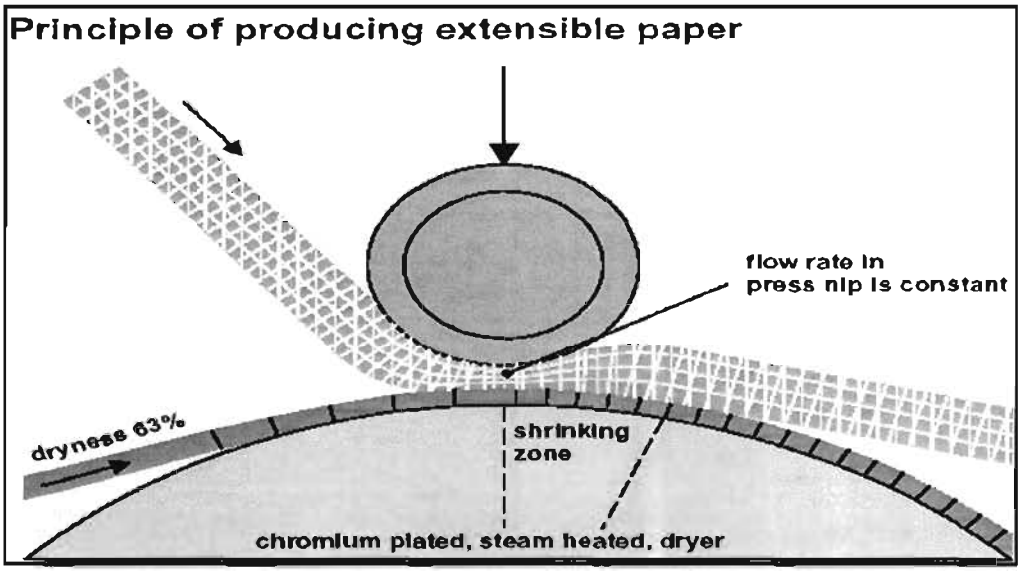



**Figure 9: Dryer cylinders operated at loose draw to enhance TEA (Fuentes *et al.*, 1981, Helle, 1998)**

Generally, HC refining is an energy- intensive operation (Karnis, 1983; Pagliarini, 1992). However, this disadvantage is compensated for by the high proportion of long fibres it preserves. HC refining causes less damage on fibre length and leaves fibres with micro - compression which improves extensibility of a paper sheet (Fahey, 1970; Gullichsen *et al.*, 1999). However, it creates curl and kinks and produces pulp with high shive content (Gullichsen *et al.*, 1999). HC refining produces pulp with a high level of fibre aggregates. It is well accepted that for commercial pulp, a system capable of dispersing those aggregates has to be implemented (Gullichsen *et al.*, 1999; Xu and Sabourin, 2002).

The pulps at HC (20-40%) are described as semi-solid. During refining, the energy is transmitted to the fibres by friction between the fibres contacting the plate surface and the surface of the fibre itself (Fahey, 1970). The coefficient of friction between refining plates and fibres has a great influence on the resulting friction forces as well as pulp quality. The friction forces are transmitted to the fibre mixture by fibre to fibre friction. The friction forces result in compression and tension stress on the individual fibres (Fahey, 1970). Rubbing, twisting and bending of the fibres break intra-fibre hydrogen bonds resulting in hydration, external and internal fibrillation (Figure 10). HC refining results in very little fibre cutting or fines formations as it has been demonstrated by Fuentes *et al* (1981), Gullichsen *et al* (1999) and most recently by Gurnagul *et al* (2005). This is in contrast to low consistency refining where fines formation is part of the process. It is speculated that LC refining results in both breakages of hydrogen bond and the covalent bond. Breakage of the covalent bond is associated with formation of fines (Fahey, 1970). Figure 10 shows a HC refiner operating principle and some refining effects on a fibre mat during HC refining.

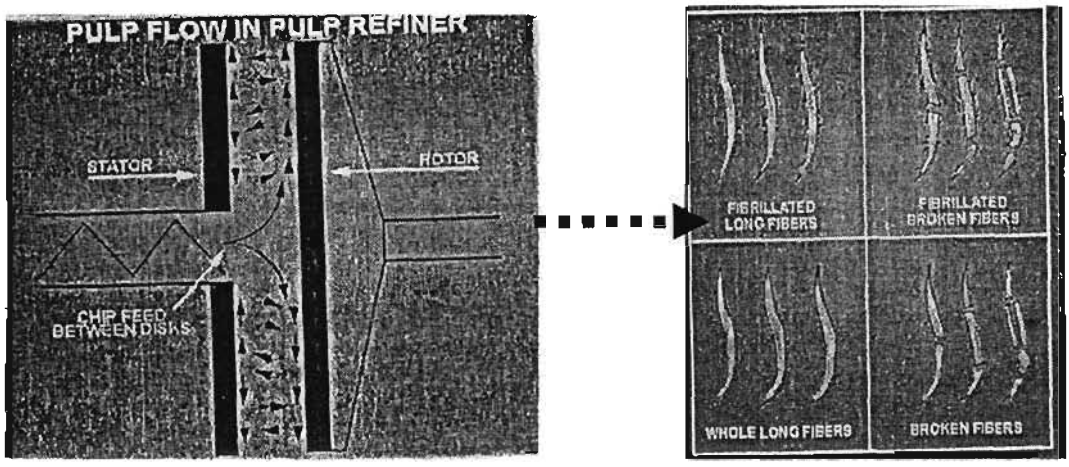



**Figure 10: HC refining mechanisms (Fahey, 1970; Jarkko, 2005)**

### 2.8.2 Low consistency refining process

LC refining is a refining operation in which the pulp suspension is fed into the refiner at about 3-4% dry solids content using a pump. Its advantage is that it removes shives and minimizes curls and kinks formed during HC refining. Figure 11 shows unrefined pulp fibres and the impact of HC followed by LC refining on the fibre morphology of the pulp.

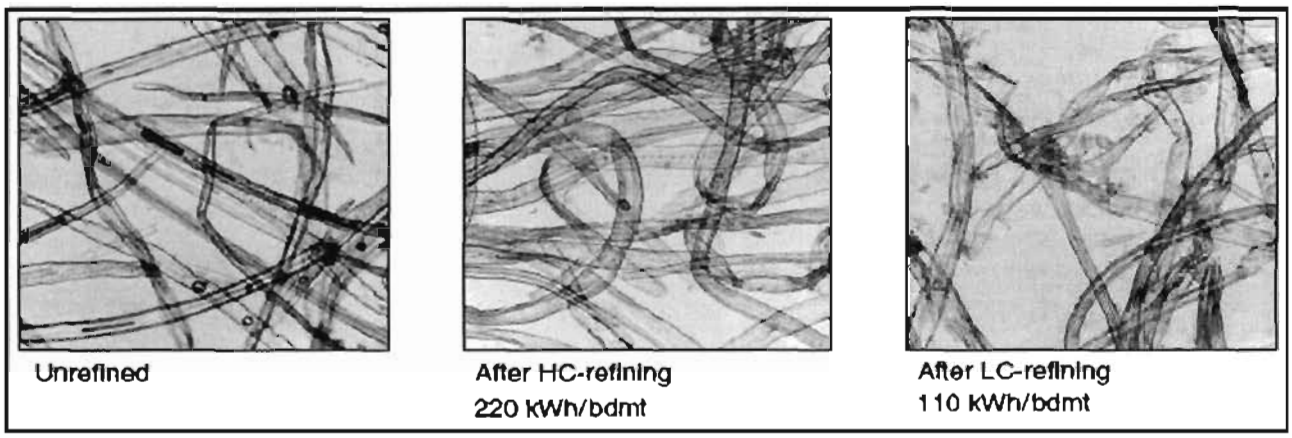



**Figure 11: Impact of HC/LC refining on fibre morphology (Gullichsen *et al.*, 1999)**

LC refining starts with fibres picked up between the stator and rotor bars (Lumiainnen, 1995). This is followed by edge to edge action ending up with surface to surface action. After the surface to surface action, the fibres are released (Figure 12).

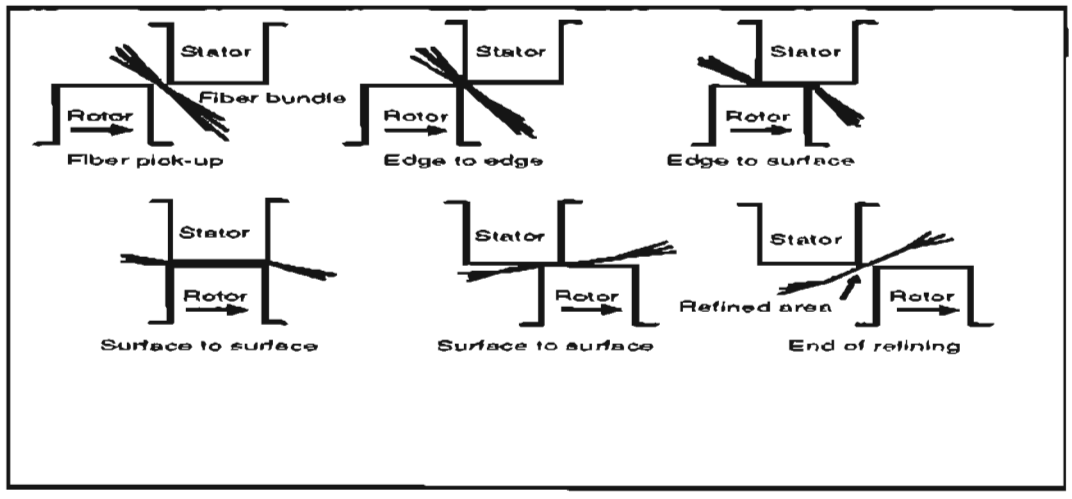



**Figure 12: LC refining mechanism (Lumiainnen, 1995)**

### 2.8.3 Impact of refining on the fibre structure

The primary cell wall which covers the fibre surface is lignin rich (Figure 3 in section 2.4.2). Lignin is hydrophobic by nature and has a lower bonding ability (Stephenson, 1950; Helle, 1998). Pulping and refining are intended to remove the restriction on the fibre cell wall and make the fibres more flexible. Flexible fibres form strong inter-bonds, resulting in high strength paper. In high yield pulping lignin is either only softened or partially removed from the fibre cell wall. High yield Kraft pulp contains large amount of lignin. Refining breaks fibre cell walls and some of the lignin fragments are removed from the fibre through leaching (Lindstrom *et al.*, 1978; Li and Macleod *et al.*, 1992).

High yield Kraft pulp fibres tend to be more flexible compared to fibres produced by purely mechanical refining of wood (Karnis, 1983; Helle, 1998). The superior bonding potential of HYK pulps becomes evident when fibres are refined sufficiently to allow the fibre lumen to collapse, the delamination of the cell wall, and the partial fibrillation of the surfaces (Helle, 1998). However, the removal of the outer shell of the stiff fibre should be done gently to minimize fibre cutting (Karnis, 1983; Helle, 1998).

The major changes in fibre structures during refining have been thoroughly investigated (Fahey, 1970; Lindstrom *et al.*, 1978; Ebling, 1980; Li and Macleod, 1992). High consistency refining promotes fibre to fibre action, thus enhancing the fibrillation effect resulting in a high proportion of undamadaged long fibres (Pagliarini, 1992; Karnis, 1983., Gurnagul *et al.*, 2005). During low consistency refining, the primary cell wall on the fibre surface is removed (Ebling, 1980; Helle, 1998). This results in fibres absorbing water and consequently swelling. Fines formation occurs as a result of loosened fragments from the primary cell wall (Fahey, 1970; Ebling, 1980). This is followed by the delamination of the external cell wall layers (external fibrillation on the outer surface of the $S_2$ layer). This occurs as a consequence of compression of the fibre mat between refiner bars followed by relaxation which leads to the breakage of the bonds within the fibre wall (Ebling, 1980; Helle, 1998). This results in both external and internal fibrillation. External fibrillation results in the exposure of micro fibrils on the surface of the fibres and fibre cutting (Fahey, 1970; Ebling, 1980; Helle, 1998). Severe fibre cutting affects the mechanical properties of the final paper product. These effects are more pronouncing at higher Kappa numbers (Hartler *et al.*, 1976). Internal fibrillation is described to be caused by the breakdown of fibre walls into separate lamellas which increase fibre flexibility (Ebling, 1980).

Dissolution of chemical components of the cell wall results in colloidal carbohydrate solution forming on the fibre surface (Ebling, 1980; Helle, 1998). This is described as the result of abrasion of the surface at the molecular level (Fahey, 1970). This enhances fibre bonding tendency which improves strength properties.

### 2.8.4 Refining parameters

The quantification of pulp refining effects on fibres are expressed in terms of refining intensity (RI) which measures the amount of refining per impact and the specific refining energy (SRE) which determines energy imparted to the fibres. This can be presented as follows:

- **Specific refining energy (SRE)**

Fibre strength development is strongly affected by the energy imparted to the fibres. The amount of SRE required is determined by the end user requirements of a particular paper grade (Gullichsen *et al.*, 1999; Gurnagul *et al.*, 2005). The SRE depends on net refining energy ($P_{net}$) which depends on the effective energy transferred to the pulp during refining, the consistency, the operating gap and the flow rate (Appendix B-3). In optimizing refiner operation, the challenge is always how these refining parameters can be manipulated to achieve the desired pulp quality.

- **Refining intensity (RI)**

Different refining theories which provide relative estimates of the refining intensity have been developed (Lumiainen, 1991; Lumiainen, 1995; Gullichsen *et al.*, 1999; Joris, 2004). These include specific edge load theory, the specific surface load theory and C-factor theory. All of these theories have focused on demonstrating the edge effect and are more applicable to low consistency refining (Fahey, 1970; Gullichsen *et al.*, 1999; Gurnagul *et al.*, 2005). HC refining does not involve the effect of the edge (Fahey, 1970; Joris, 2004). Little information has been published for HC refining intensity (Gullichsen *et al.*, 1999; Gurnagul *et al.*, 2005).

In an attempt to determine the effects of HC-refining variables on the RI, Gurnagul *et al.*, (2005) conducted a study to demonstrate how RI for HC refining can be evaluated with respect to its effect on pulp quality (see Appendix B-3). They found that an increase in refiner speed increases the refining intensity while an increase in pulp consistency reduces the refining intensity, provided that other variables are kept constant. However, this relationship has not yet widely been accepted for daily application in industry.

### 2.8.5 Parameters influencing pulp strength development

- **Pulp yield**

The higher the yield the stiffer the fibres (Hartler *et al.*, 1976), thus higher yield pulp requires more energy compared to pulp at lower yield. The reason is that fibres at lower yield are more flexible and readily absorb energy thus can be easily beaten (Fahey, 1970; Ebling, 1980). On the other hand, stiff fibres respond in the opposite way, requiring more energy to attain the same level of flexibility as conventional pulps. This suggests that for optimum strength development, each pulp yield level requires specific amounts of specific refining energy as well as refining intensity.

- **Machine parameters**

**No load power:** The no Load power is measured when the gap of the refiner is fully open. This does not cause a measurable change in the pulp properties and the energy used is associated with mechanical losses only (Joris, 2004). The no load power must be excluded when measuring the energy imparted to the fibre during refining. The effect associated with an increase in no load power is that it reduces the refiner capacity due to reduced net power (Stein, 1981; Joris, 2004). The higher the no load power, the less the energy imparted to the fibres (equation 7 and 8 Appendix B-3).

**Refiner operating speed**: Increasing refiner speed, increases inch-contact-per-minute which favours internal brushing (Stein, 1981; Gullichsen *et al.*, 1999). High operating speed increases the number of bar crossings and reduces refining intensity which favours the fibre fibrillation effect (Stein, 1981; Pagliarini, 1992). This phenomenon explains why high consistency refiners are characterized by a high operating speed.

**Refining plate design**: The plate pattern configuration has a great influence on the pulp quality. It has been demonstrated that wide grooves, less dams or steeper taper increases refining intensity (Stein 1981; Lumiainen, 1993; Gullichsen *et al.*, 1999; Joris, 2004; Jarkko, 2005). The angle of intersection of the crossing bars between stator and rotor (Figure 14) also affects fibre strength development (Stein, 1981; Joris 2004). It is speculated that an increase in the angle between the rotor and the stator increases the power; however, fibre cutting is reduced (Fahey, 1970; Backer, 1994). The effect of grinding code (bar width, groove width and groove depth) are illustrated in Figure 13. Wider bars with the same groove width favours fibrillation while a plate with a narrow bar width and wider grooves favours fibre cutting.
The selections of the refiner fillings are governed by fibre type, refining conditions as well as refining target. For long and strong fibres of high yield softwood Kraft pulp wider bars and wider grooves are required (Backer, 1994; Gullichsen *et al*, 1999).

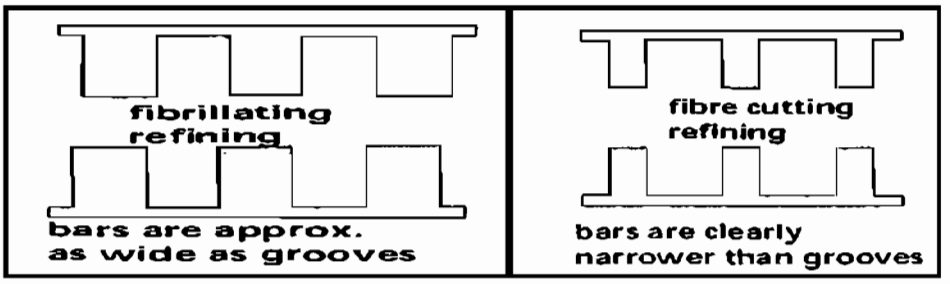



**Figure 13: Refiner plate with grinding code for different refining purposes (Lumiainen, 1993; Lumiainen, 1995)**

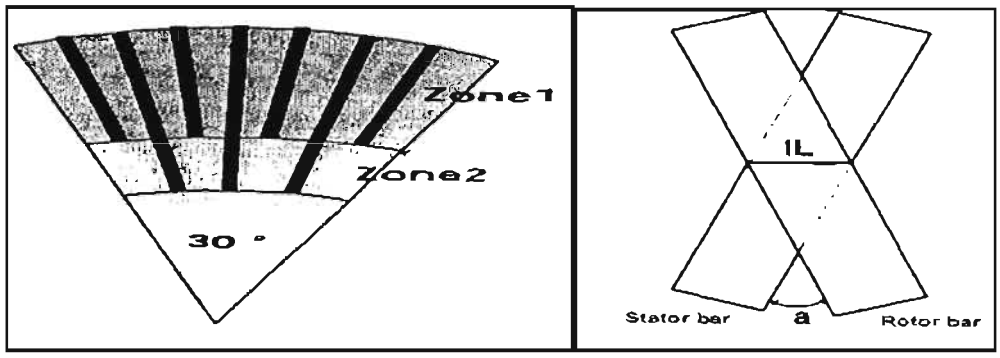



**Figure 14: Illustration of how sector angle (30°) and grinding angle (a°) for a refiner plate is defined (Lumiainen, 1995)**

**The refiner tackle materials**: The effects of refiner tackle construction materials have an effect on pulp quality and refiner operation (Fahey, 1970; Gullichsen *et al.*, 1999; Joris, 2004). If different materials are used to refine the same pulp, it produces pulp with different pulp quality (Fahey, 1970). A comparison of steel, bronze and lava tackle has shown that pulp refined with lava tackle produces pulp with a higher tear, elongation, bulk and porosity (Fahey, 1970; Helle, 1998). Higher breaking length and burst where obtained using steel bars, and the lowest value was from bronze bars (Fahey, 1970; Helle, 1998). The fibre quality is affected by the deterioration of the leading edge of the refiner plate bars as a result of wear, corrosion, cavitation or breakage of edge of the refiner plate bars. It is also well understood that by operating a refiner with worn bar edges, more energy is required to maintain the same pulp quality (Fahey, 1970; Gullichsen *et al.*, 1999). Therefore, choosing the right blend of alloy elements that make up the plate is of paramount importance. There are two categories of refiner plate alloys, namely white iron and stainless steel. The major distinction between these two alloys is that white iron contains a high content of chromium and carbide compared to stainless steel. Thus white iron has excellent wear and corrosion resistance, but inferior breakage resistance, due to the high carbide content which makes the alloy very hard but exceptionally brittle.

**Gap clearance**: The gap clearance is the distance between the opposing rotor and stator bars. The refiner operating gap has an influence on both refining intensity and the specific refining energy (SRE) as was demonstrated by Karnis *et al.*, (1991). In this study it was found that a small gap clearance favours high refining intensity and increased SRE, while increasing gap clearance favours lower refining intensity and lower SRE, given that the other variables are kept constant. The size gap should be set up depending on SRE target or intensity requested, which is determined by the pulp quality desired.

- **Pulp consistency**: Pulp consistency is the measure of amount of dry solids in a given pulp mass. The pulp refining consistency has a positive influence on fibre strength development. However, high pulp consistency above 40% may lead to plate plugging resulting in operational problems (Gullichsen *et al.*, 1999). In an attempt to explain the importance of high consistency refining on fibre strength development, Page (1971) did a comparison between HC and LC refining. Initially he found that HC refined pulp showed higher TEA. On inspection of the fibres, it was found that HC refined pulp fibres had a closely packed structure of compressed regions along the entire length of each fibre. He suggested the partial recovery from this micro compression to be responsible for increased TEA for paper made from high consistency refined pulp. In addition, he also found that fibre length was only reduced by a small percentage. These findings suggested that HC refining enhances the extensibility of paper with high tear strength due to less fibre cutting. These findings by Page were also reported later by Fuentes *et al.*, (1981), Pagliarini (1992), Gullichsen *et al.*, (1999) and most recently by Gurnagul *et al.*, (2005).

- **Feed rate**: Stock feed rate is a measure of the material flow into the refiner. The stock feed rate influences the energy transferred to the stock and thus the pulp quality (Karnis *et al.*, 1991). Flow rate can be used to alter both SRE and RI provided that other processing variables are kept constant. Fluctuations in flow rate results in uneven refining resulting in variations in pulp quality and can also lead to operational problems.

- **pH**: It has been demonstrated that pulp pH has an effect on fibre strength development during HC refining (Helle, 1998). High pulp pH accelerates fibre cutting and the effect is more pronounced at high Kappa numbers (Helle, 1998). Pulp pH during HC refining should be kept close to 8 (Helle, 1998). This is in contrast to LC refining, where faster refining is enhanced by fibre swelling which is more pronounced at pH> 7 (Helle, 1998; Per- Michael, 2002).

- **Refining zone temperature**: Refining at high consistency results in temperature build-up and shear forces in the refining zone (Senger *et al.*, 2002). The refining zone temperature affects the equivalent tangential coefficient of friction in the refiner. An increase in temperature strongly affects the visco-elastic properties of the pulp. At high temperature, low visco-elasticity corresponds to lower equivalent tangential coefficient of friction. Consequently the shear forces acting on the pulp mat in the

refiner are lower. The shear forces are speculated to be responsible for the external fibrillation of the fibre wall during the HC refining operation (Senger *et al.*, 2002). The temperature range is limited to 180 °C for industrial refiners while for pilot refiners the temperature should be kept at 100 °C (Senger *et al.*, 2002).

### 2.8.6 Types of refiners used for high consistency refining

The most common refiners used for high yield pulp are the single disc refiners and occasionally double disc refiners and conical refiners (Kurdin, 1997; Gullichsen *et al.*, 1999). Figure 15 and 16 illustrate types of refiners commonly used in stock preparation of high yield pulp.

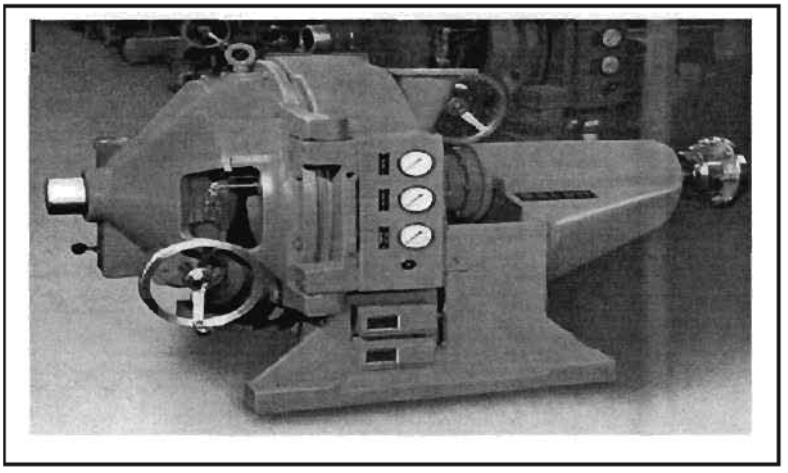


**Figure 15: Typical Beloit Double Disc refiner (Gullichsen *et al.*, 1999)**

The low capacity and difficulties in change of fillings of the conical disc refiners have limited their application in industry (Lumiainnen, 1991; Gullichsen *et al.*, 1999).

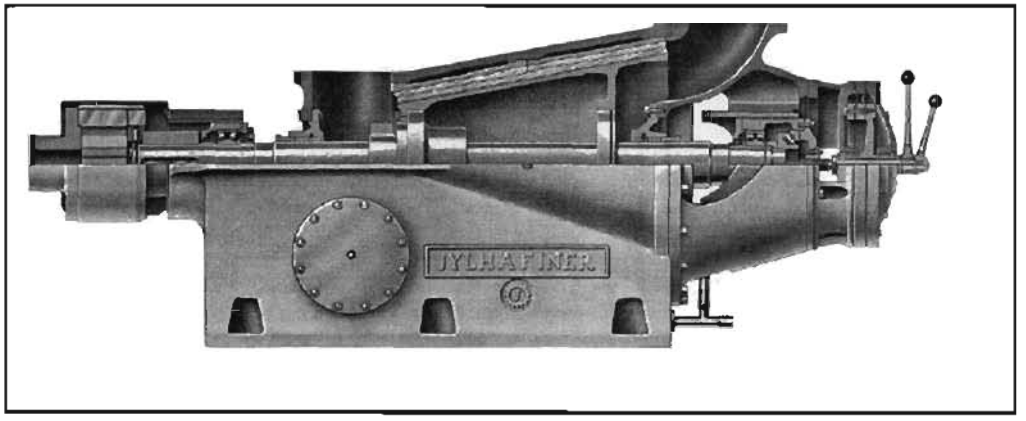



**Figure 16: Cross section of Jordan-type conical refiner (Gullichsen *et al.*, 1999)**

### 2.8.7 Refining approach to optimize high yield pulp quality

In any paper mills, refining configuration depends on many factors. These include the nature of the wood species to be used, product quality requirements etc. Refining configuration may be HC refining, LC refining, or a hybrid process of the two (HC combined with LC) as primary and secondary refining respectively (Hgemeyer and Mason, 1992; Gullichsen *et al.*, 1999; Xu and Sabourin, 2002). In this study the focus was on whether HYK with Kappa number higher than 70 can develop strength properties suitable for sack paper grade. A hybrid process combining HC and LC refining was adopted. Several studies have investigated the benefits of second stage refining by LC refining (Gullichsen *et al.*, 1999; Xu and sabourin, 2002). Table 1 illustrates some of the effects of HC and LC refining configuration on the handsheet quality properties. Figure 17 shows a typical HC and high yield industrial refining system.

**Table 1: The important effects on handsheets due to HC and LC refining (Ebling, 1980; Hell, 1998; Gullichsen *et al.*, 1999)**

| Pulp properties | HC refining stage | LC refining stage |
|---|---|---|
| Uniformity | Poor | Good |
| Formation | Poor | Good |
| Smoothness | Poor | Good |
| Density | Low | High |
| Air flow resistance, | Low | High |
| Bending stiffness | High | Low |
| Sheet shrinkage | Good | Improved |
| Fines formation | Low | High |
| Bulk | High | Low |
| Curl and Kinks | High | Low |

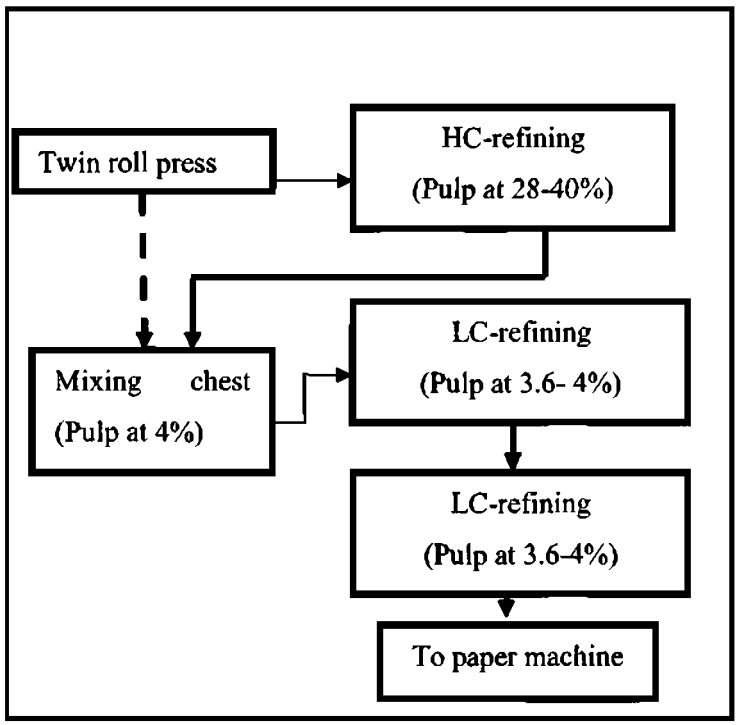



**Figure 17: Typical HC+LC refining in a high yield industrial refining system**

**(Gullichsen *et el*, 1999)**

# CHAPTER 3: COMPARISON BETWEEN INDUSTRIAL AND LABORATORY REFINING

## 3.1 Introduction

A comparison between industrial and laboratory (HC+LC) refining was performed to get an understanding of whether laboratory refiners at the FFP Laboratory could be used to simulate an industrial refining processes. This could help in optimizing the experimental refining conditions for the subsequent work in this project.

## 3.2 Material and methods

### 3.2.1 Material

Pulp samples were collected from Sappi Tugela mill before and after the high consistency refiner (HCR) and after the low consistency refiner (LCR). The pulp Kappa number was 70.

### 3.2.2 Experimental design

The experimental design for comparison between laboratory and mill refining is illustrated in Figure 18. Batches of industrial pulp at Kappa 70 were sampled after a double wire press at Sappi Tugela mill (sample 1) were refined by laboratory refiners (HC+LC) and the pulp quality in terms of strength properties and fibre morphology were compared with mill HC+LC refined (samples 2 and 3) as shown in Figure 18.

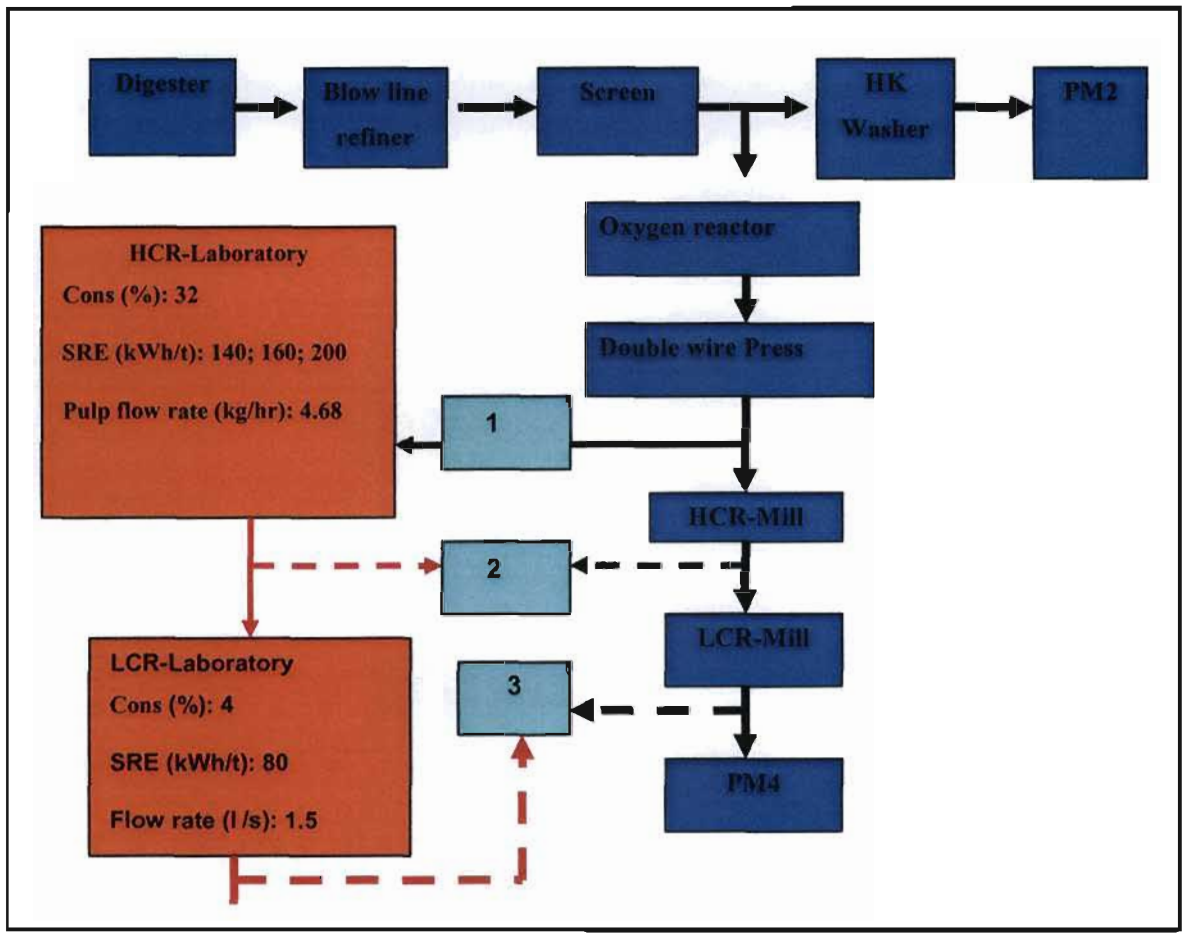



**Figure 18: Experimental design for comparison between industrial and laboratory refiners (HCR: high consistency refiner, LCR: low consistency refiner)**

### 3.2.3 Equipment

- **High consistency refiner**

Prior to the start of the experimental work, HC refiner operating conditions were optimized as detailed in Appendix B-1. The laboratory high consistency refiner is a single disc 12" inch Sprout Bauer refiner (atmospheric discharge). The capacity of the refiner is 37 kW with an operating speed of 3149 rpm. The operating speed of the refiner was verified by using a tachometer. The refiner operating speed can be changed by adjusting the pulley and V-belt system. The model of plate used was 2A501. The refiner was provided with a calibrated gap adjustment wheel, which was used to set up the operating gap between the stator and the rotor. Table 2 shows the plate pattern specification and the plate pattern used is illustrated in Appendix B-2. The flow diagram for the laboratory HC refiner and the pulp feeding system layout is illustrated in Figure 19.

**Table 2: HCR Plate pattern specification for the plate model 2A501**

| Plate specifications | Values |
|---|---|
| Bar width (mm) | 3 |
| Groove width (mm) | 3 |
| Groove depth (mm) | 4 |
| CEL (km/rev) | 2.27 |
| Cutting speed (km/s) | 119 |
| Material | ** Stainless steel |

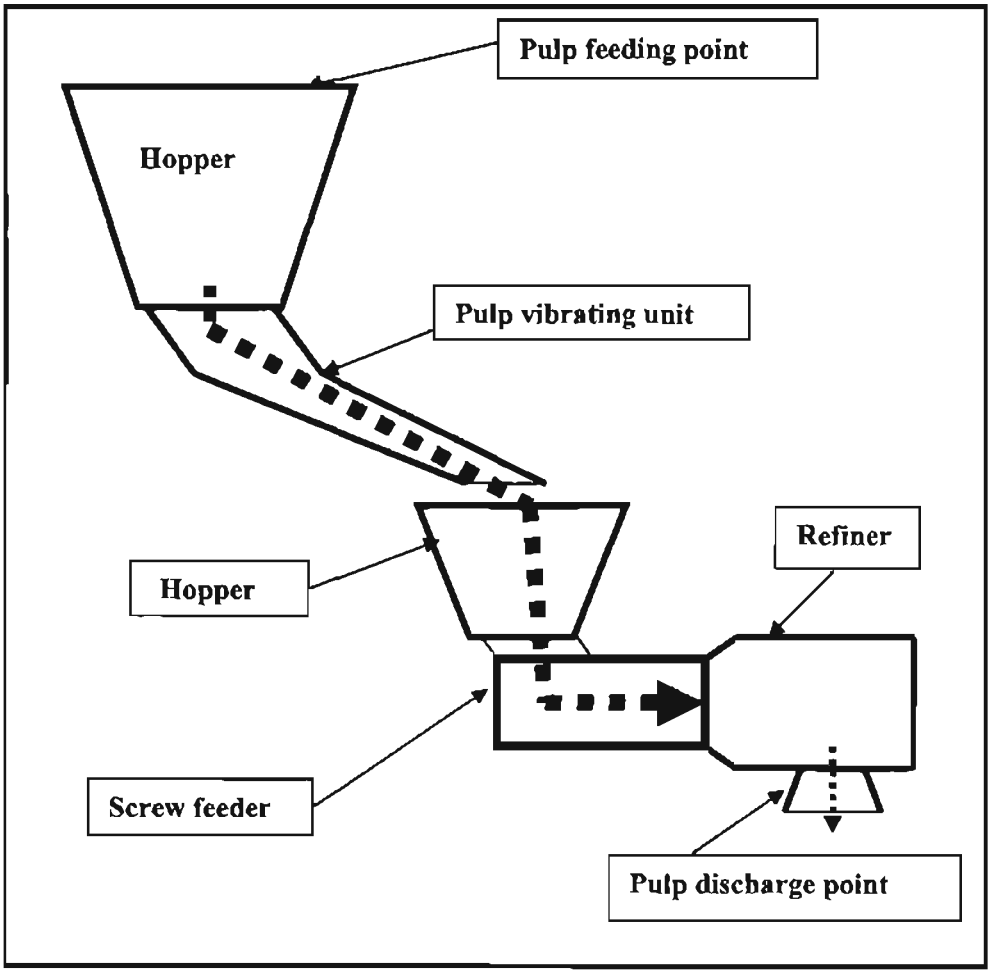



**Figure 19: Flow diagram of the laboratory HC refiner**

This plate model was designed to handle pulps of up to 25% consistency and the trial required feeding of pulp up to 35% consistency. A series of trials were conducted to determine the

maximum operating consistency. It was found that it was possible to operate up to 32% consistency without plate clogging. Higher consistency was preferred because the higher the consistency the better the fibrillation effect (Fahey, 1970; Gullichsen *et al.*, 1999; Gurnagul *et al.*, 2005). The pulp mass flow rate into the refiner is a critical variable, not only for achieving good pulp quality, but also for ensuring smooth operation (avoid screw feeder jamming and plate plugging). Optimum operating mass flow rate was first established. It was found that for smooth operation of the refiner, the screw feeder speed must be kept at 85 rpm. This speed gives a pulp mass flow rate of 4.68 kg/hr (bone dry mass). This flow rate was used for all trials in this project.

The no load power was determined by running the refiner in full open position without pulp. The gap clearances at which the target SRE could be achieved were also established. This was done by running the refiner at different gap sizes and recording the gross power (Karnis *et al.*, 1991). The values of no load power, gross power, consistency and pulp mass flow rate were then used for calculating SRE (See sample calculation in Appendix B-3). After determining the correct gap setting for each target level of SRE, three trials were performed at three levels of SRE. Table 3 shows the average SRE for the different gap sizes. These gap settings were used for all trials in this project. When a higher SRE was required, several passes were performed either at one gap setting or at more than one of these gap settings, depending on the target SRE required.

**Table 3: Gap size and mean SRE during the HC refining trials**

| Gap size (microns) | Average SRE (kWh/t) | Standard deviation |
|---|---|---|
| 625 | 140 | ±0.18 |
| 500 | 160 | ±0.20 |
| 375 | 200 | ±0.38 |
| 350 | 240 | ±0.40 |

- **Low consistency refiner**

The LC refiner is a single 8" (20cm) disc Sprout Bauer refiner (pressurized refiner). The capacity of the refiner is 22 kW with a variable speed drive. The refiner rig consists of two agitated tanks (with capacity of 400 l each). During operation, the pulp suspension was transported at the desired consistency from one tank to the other through the refiner by pumping with a positive displacement pump (a progressive cavity pump or mono pump). The

pulp flow was monitored by an electro-magnetic flow meter (flowmag) fitted on the refiner piping system. The target SRE was achieved by varying the number of passes. Pulp was sampled for each pass through the sampling valve situated after the refiner. Table 4 shows the specification of the plate pattern used for LC refining. The picture of plate pattern used is illustrated in Appendix B-2. The process flow diagram for the laboratory LC refiner is shown in Figure 20.

**Table 4: Plate pattern specification for LCR**

| Plate specification | Value |
|---|---|
| Bar width (mm) | 5 |
| Groove width (mm) | 5 |
| Groove depth (mm) | 6 |
| Grinding angle (degrees) | 10 |
| CEL (km/rev) | 0.18206 |
| Material | ** Stainless steel |

** The plate pattern made of stainless steel alloy was preferred in this study because it produces pulp with superior quality compared to other alloys. In addition to that it has a low bar edge deterioration rate (Helle, 1998), and thus has more life time.

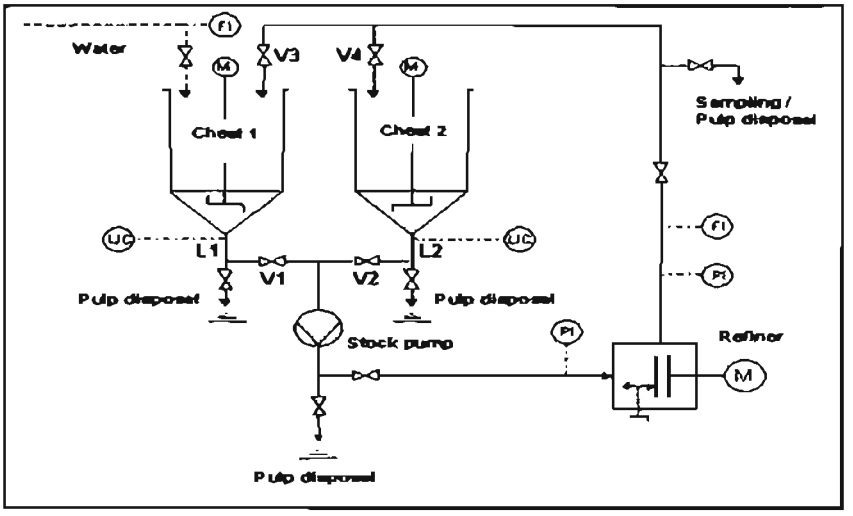



**Figure 20: Process flow diagram of the laboratory LC refiner**

The no load power was determined by running the refiner with only water at the operating speed with the refiner gap at maximum (full open). A pulp mass equivalent to 10 kg oven dry (OD) pulp was charged to the refiner chest for each trial. Each pulp sample was then diluted

to the desired consistency (4%), before refining (see dilution sample calculation in Appendix B-3). The operating gap was set by adjusting the gap up to gross power reading of 27% (5.94 kW). This value was used for all trials in this project as a reference for the operating gap setting. This was followed by determining the number of stages required to achieve a target SRE. This was done using pulp volumetric flow rate, pulp consistency, no load power and the gross power attained at various refining stages (see sample calculation in Appendix B-3). Table 5 shows the results for SRE at various refining stages which were used during LC trials in this project. Four repeats were performed.

**Table 5: Raw data collected during the LC refining trials**

| No of stages / passes | Target SRE (kWh/t) | Standard deviation |
|---|---|---|
| 7 | 80 | ± 1.8 |
| 10 | 120 | ± 2.2 |
| 15 | 180 | ± 3.3 |
| 17 | 200 | ± 2 |

## 3.3 Testing of pulp quality

### 3.3.1 Freeness

After refining the HYK pulp, the Canadian Standard Freeness (CSF) measurement was performed according to TAPPI test method T227 om-94 (Xu and sabourin, 2002; Gurnagul *et al.*, 2005).

### 3.3.2 Preparation of handsheets

Hand sheets with a grammage of approximately 60 g/m$^2$ were prepared according to T205 sp-95 for testing of physical properties. The pulp suspension left in the stock divider after the freeness test was used for hand sheet making for each sample. Prior to hand sheet forming, a test sheet was made to determine the consistency of the pulp suspension in the stock divider. This is because, to form a hand sheet with a basis weight of about 60 g/m$^2$ on the hand sheet former with an area of 31400 mm$^2$, the acceptable paper sheet weight range is in the range of 1.885-1.98 g.

Ten hand sheets were made for each sample using the rapid Kothen machine. Blotting paper (one placed between each sheet and three placed on the top and bottom) were used as felts.

This is because when a wet sheet is placed between felts and pressed, the felt absorbs the water (Hell, 1998), thus raising sheet solid content as well as the initial wet strength. The sheets were further pressed at 600 kpa for five minutes to remove excess water. Each sheet was then dried in a speed drier for four minutes. Hand sheets with a grammage of approximately 60 g/m$^2$ were prepared according to TAPPI test method T205 sp-95 for testing of physical properties (Xu and sabourin, 2002, Gurnagul *et al.*, 2005). The prepared hand sheets were left in the conditioning room for 24 hours prior to testing. The room was kept under standard conditions (at 23 °C ± 0.5 °C and relative humidity 50% ± 1).

### 3.3.3 Testing of sheet physical properties

TAPPI standard methods were used for all tests that were performed. Table 6 summarizes the pulp physical properties tested with the corresponding TAPPI test standard methods used.

**Table 6: Pulp strength properties tested and test methods used (TAPPI test method, 2000; Xu and sabourin, 2002; Gurnagul *et al.*, 2005).**

| Standard Testing method | Pulp quality properties tested |
|---|---|
| T 220 sp-96 | Basis weight |
| T 494 om-88 | Tensile strength, TEA, Stretch |
| T 414 om -88 | Tear strength |
| T403 om-91 | Burst strength |
| T547 pm-95 | Porosity |
| T 231cm-96 | Zero span tensile strength |
| T556pm-95 | Bending stiffness (at bending angle of 15°) |

### 3.3.4 Fibre morphology

Fibre morphology was analyzed using the Techpap MorFi fibre analyser (v 7.9.13 [MTL 7.5-Sp1C/NuDAQ 3.0]) at the Sappi Technology Centre. This instrument uses a camera and image analysis software to characterize the fibre morphology (fibre length, shives and fine elements through size criteria). The analysis was done at a consistency of 30 mg/l. Table 7 summarizes the pulp fibre morphology measured on the MorFi and the size limits that were set for each of the characteristics measured.

**Table 7: The pulp fibre morphological properties and the size limits for each of the fibre characteristics analyzed**

| Parameter measured | Specification |
|---|---|
| Fibre length | 200 µm< fibre length < 10 000 µm |
| Fibre width | 5 µm < fibre width < 75 µm |
| Fines | fines length < 200 µm |
| | fines width < 5 µm |
| Shives | 200 µm< fibre length < 10 000 µm |
| | fibre width >75 µm |
| Curl and kinks | Percentage of curled or kinked fibres |

## 3.4 Results and discussion

The values for strength properties which were attained in these trials are illustrated in Table 8. It can be seen that the laboratory refined sample at 200 kWh/t using the HC refiner and further refining at 80 kWh/t with LC refiner produced pulp with qualities comparable to the mill refined samples. Thus all comparisons refer to laboratory samples refined at 200 kWh/t in the HC-refiner.

The freeness of the laboratory HCR samples dropped from 685 to 668 ml (17 units) while freeness for industrial HCR samples dropped from 685 to 620 ml (65 units). The difference in drop of freeness values between mill and laboratory samples was statistically significant ($p=0.03$). The strength values after HC laboratory refiner were lower compared to pulp strength after HC mill refiner. This was expected since the freeness of the pulp after HC refining in the laboratory was significantly lower.

**Table 8: Comparison of pulp strength properties after HCR between mill and laboratory refiner for Kappa 70 pulp**

| Refining stage | | Laboratory samples | | | | | | Mill samples | |
|---|---|---|---|---|---|---|---|---|---|
| | *Unrefined* | *HCR* | *LCR* | *HCR* | *LCR* | *HCR* | *LCR* | *HCR* | *LCR* |
| SRE (kWh/t) used | | 140 | 80 | 160 | 80 | 200 | 80 | 180-200 | 80 |
| Freeness (ml CSF) | 685 | 681 | 603 | 678 | 590 | 668 | 575 | 620 | 560 |
| Tensile index (KNm/kg) | 42 | 44 | 55 | 48 | 64 | 52 | 66 | 60 | 73 |
| Tear index ($Nm^2/kg$) | 11 | 10 | 7.8 | 9.7 | 7 | 9 | 7.7 | 10.5 | 8 |
| Burst index (mN/kg) | 2.5 | 3.4 | 4.8 | 3.8 | 5.2 | 4.1 | 5.7 | 5.2 | 6.3 |
| TEA index (kJ/kg) | 0.8 | 0.9 | 1.4 | 1 | 1.5 | 1.32 | 1.6 | 1.5 | 1.7 |
| Stretch (%) | 2.5 | 2.26 | 3.28 | 2.73 | 3.33 | 2.96 | 3.43 | 2.8 | 3.74 |
| Porosity (Gurley s/100ml ) | 0.9 | 1 | 4.5 | 1.6 | 5.5 | 2 | 7.2 | 2 | 8 |
| Bending stiffness (mN) | 62 | 55.3 | 49 | 55 | 45 | 46 | 42 | 48 | 42 |
| Zero span tensile strength (N) | 41 | 42 | 57 | 45 | 60 | 59 | 62 | 63 | 62 |
| Fibre length (mm) | 2.22 | 2.28 | 1.59 | 2.14 | 1.97 | 2.28 | 1.94 | 2.18 | 2.11 |

In samples refined using 200 kWh/t SRE for HCR then 80 kWh/t for the LCR, the freeness dropped from 668 to 575 ml (93 units) while freeness for industrial samples dropped from 620 to 560ml (60 units). The difference in freeness mean values between laboratory and mill LC refining were not significant (p=0.4). This was supported by similar values of strength properties between mill and laboratory after LC refining (Table 8). Generally, it was observed that after the LC refining stage most of the strength properties improved except tear and bending stiffness. This trend explains the importance of having HC and LC refining stages in a high yield pulp refining system.

Comparing quality parameters measured in sack Kraft paper, it can be seen that mill samples exhibited slightly higher TEA and stretch while bending stiffness was similar. Higher TEA for mill samples could be attributed to the different refiner plate pattern found in the industrial refiner compared to that of the laboratory refiner. The laboratory HC refiner plate pattern could only handle pulp with consistency 25-32% while HC mill plates operate at a higher consistency (36-40%). It is well accepted that the higher the consistency the better the stretch which enhances TEA development (Fahey, 1970; Pagaliarini, 1992; Gurnagul *et al.*, 2005). Fibre curl and kink before and after HC and LC refining were evaluated (Figure 21 and Figure 22). The number of curled fibres was slightly higher after HC laboratory refining compared to pulp after HC mill refining (Figure 22). This could be attributed to the mill process; after

HC refining, pulp is diluted and mixed thoroughly. This may reduce the number of curled fibres (note: laboratory samples are sampled when they are still in fibre network/mat form).

It is speculated that fibre curl induced during HC refining enhances extensibility of paper (Kibblewhite *et al.*, 1979; Gurnagul *et al.*, 2005). The paper shrinkage potential depends on extensibility (Gurnagul *et al.*, 2005). Curled fibres have a beneficial effect on paper grades that require a high level of extensibility (stretch) such as sack paper grades. However, excessive curl affects fibre strength development (Fuentes *et al.*, 1981; Gullichsen *et al.*, 1999). The effect of curl after HC refining is minimised with LC refining (Figure 21). In numerous studies, it has been reported that LC refining improves bonding ability through reducing fibre curl and kink (Gullichsen *et al.*, 1999; Gurnagul *et al.*, 2005). In these trials both laboratory and mill LC samples showed similar percentages of curled fibres.

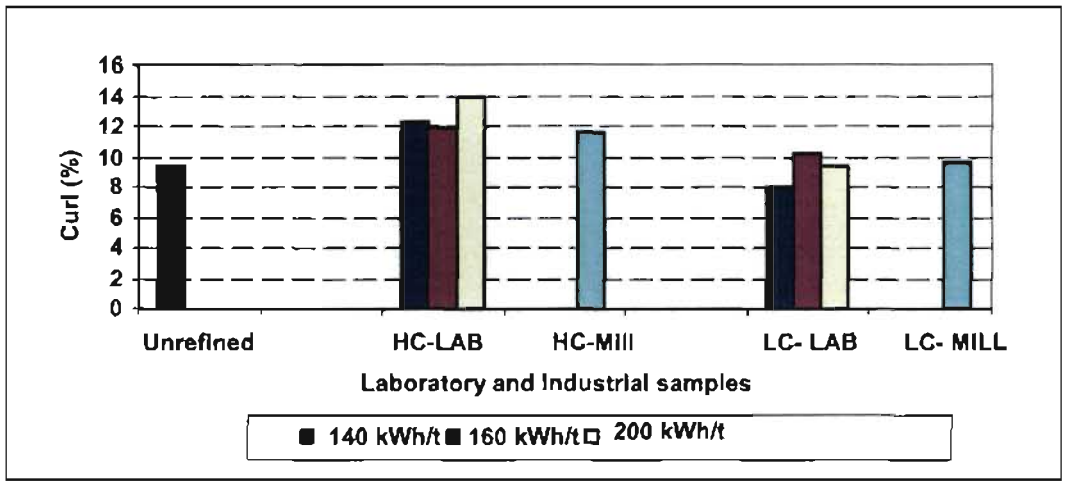



**Figure 21: Impact of laboratory and mill HC and LC refiner on percentage of curled fibres**

Kinked fibres refers to fibres with sharp bends as a result of the effect of compression and interaction of fibres during the refining process. The results indicated that laboratory HC refined samples have a higher percentage of kinked fibres compared to mill HC samples, and the explanation is the same as given for curl. The presence of these deformations leads to weak points on fibres, thus having negative effects on strength properties (Page, 1971). After LC refining the number of kinked fibres was reduced. However, the laboratory sample exhibited slightly higher percentages of kinked fibres (Figure 22). Figure 22 shows that the percentage of kinked fibres after HC refining was significantly reduced after LC refining for both mill and laboratory sample ($p=0.03$). This shows the importance of LC stage in HYP refining system (Gullichsen *et al.*, 1999; Xu and sabourin, 2002). Reduction of curl and kinks improves development of strength properties (see table 8).

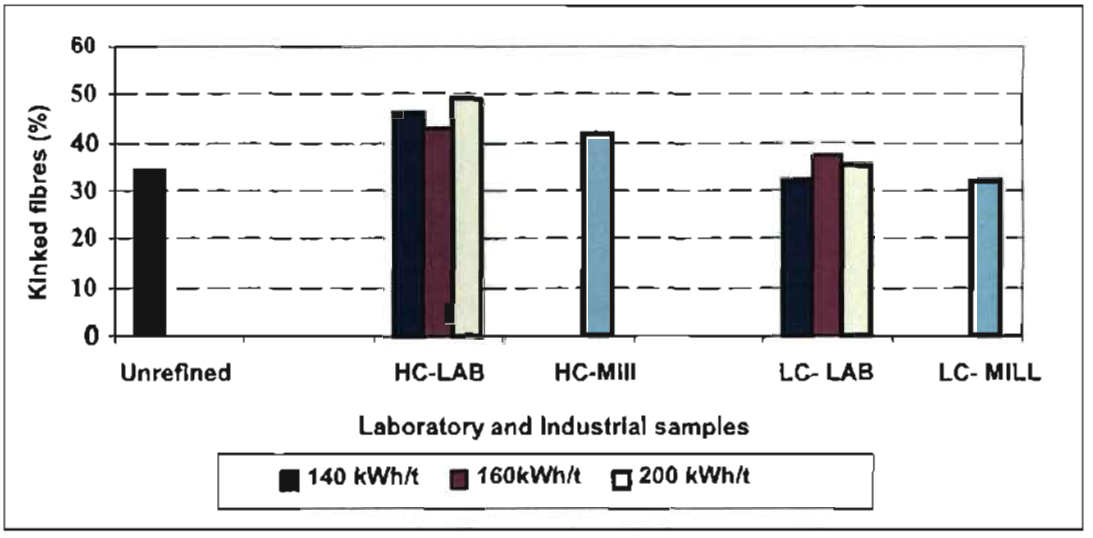



**Figure 22: Impact of laboratory and mill HC and LC refining on percentage of kinked fibres**

## 3.5 Conclusions

The focus of this work was to identify the ability of the FFP laboratory HC and LC refiners to simulate industrial refining processes. The results indicated that the laboratory refined pulp samples at 200 kWh/t SRE for HC refining followed by 80 kWh/t SRE LC refining produces similar pulp properties to those currently achieved at Sappi Tugela mill. These results suggest that under well-controlled refining conditions, the pilot scale laboratory refiners at the FFP laboratory can be used to simulate industrial refining processes.

All of the test values of TEA were lower compared to target commercial value of 2.2 kJ/kg (Gurnagul *et al.*, 2005). These results suggest that the laboratory HC refiner did not induce adequate stretch which is responsible for TEA development (Pagliarini, 1992; Gurnagul *et al.*, 2005). This may be attributed to plate pattern which had the limitation of refining only 25-32% consistency pulp. The HC refiner should operate at a higher consistency (36-40%) to improve stretch. TEA is a critical quality property for sack paper, and thus one of the most significant quality parameters to mills such as Sappi Tugela mill.

Results showed that after the LC refining stage most of the strength properties improved to an acceptable level, except tear and bending stiffness. This shows the importance of the LC stage in improving strength of high yield pulp.

# CHAPTER 4: COMPARISON OF STRENGTH PROPERTIES OF INDUSTRIAL PULP AT KAPPA 70 AND 85

## 4.1 Introduction

The focus of this element of the work was to understand how the strength properties of high Kappa number pulps can be optimized. The influences of Kappa number and SRE on strength development based on industrial high yield pulps currently produced in industries were studied. An understanding of these effects was essential in order to determine energy input in terms of SRE which can provide optimum pulp strength development for pulp with higher Kappa numbers in phase three trials.

## 4.2 Material and methods

### 4.2.1 Material

Pulps at Kappa 70 were sampled from Tugela mill before the HCR. Pulps at Kappa 85 were sampled after the high Kappa washer in the same pulp mill.

### 4.2.2 Experimental design

The experimental design for these trials is shown in Figure 23. The pulp samples after being refined by laboratory refiners (HC+LC), the pulp quality in terms of strength properties and fibre morphology for pulp at Kappa 70 and 85 were compared.

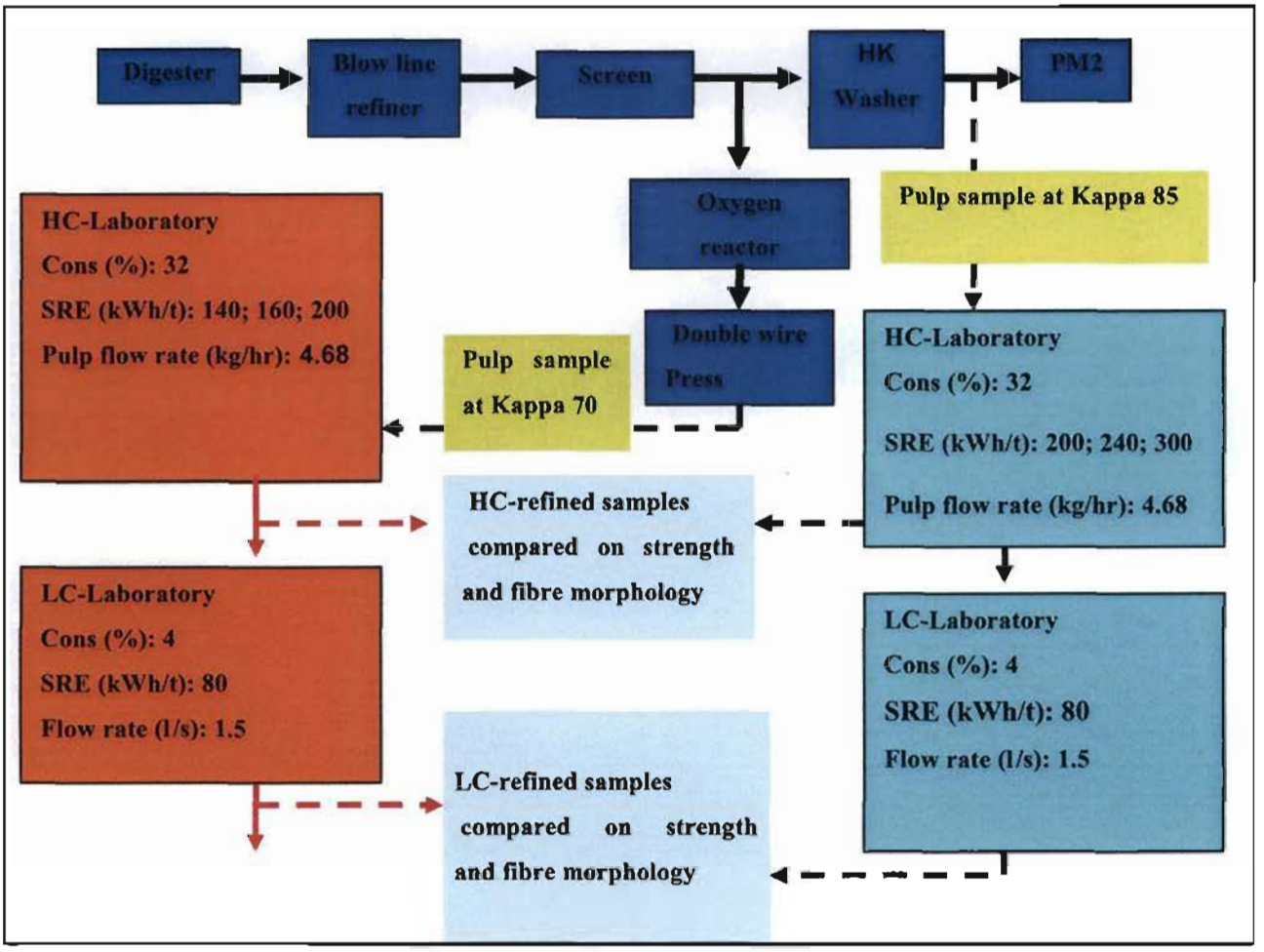



**Figure 23: Experimental design for the refining trials for pulp at Kappa number 70 and 85**

### 4.2.3 Refining procedures

The same methods described in Chapter three trials were used in this study.

### 4.2.4 Testing of pulp quality

The same procedures described in Chapter three (section 3.3) were used.

## 4.3 Results and discussion

### 4.3.1 Freeness

Freeness is a very important parameter because it influences paper machine performance (Helle, 1998; Xu and sabourin, 2002). Figure 24 shows the relationship between freeness and SRE for the two different Kappa number pulps after HC and LC refining. The freeness of pulp at Kappa 85 was higher by 23 units compared to pulp at Kappa 70.

For all HC refined samples, freeness decreased as SRE increased. This is because a higher SRE makes the fibres more flexible and collapsible. Due to higher flexibility, the fibre mat is denser and drainability decreases (Ebling, 1980; Helle, 1998).

The evaluation of the effect of HC refining on freeness drop indicated that pulp at Kappa 85 exhibited lower freeness drop (four units) while pulp at Kappa 70 dropped 17 units at the same SRE. This difference in freeness drop between the two pulps was significant (p=0. 001). This may be due to increased fibre stiffness at higher Kappa number (Hartler *et al*, 1976). Stiff fibre does not conform or consolidate well and as a result leaves more pores open which facilitate drainage (Helle, 1998).

After LC refining stage, freeness for both Kappa numbers was further reduced in comparison to the values obtained after HC refining (Figure 24). It was observed that the values of freeness for Kappa number 85 were slightly higher compared to pulp at Kappa 70 (by 4.5% at the same SRE level). This showed that LC refining did improve the fibre flexibility. However, from Figure 14, LC refining between the two pulps was not significant (p=0.8).

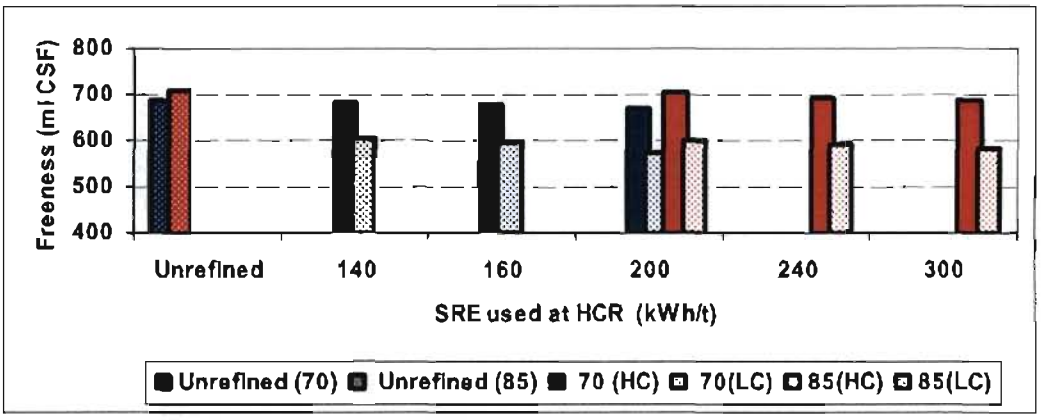



**Figure 24: Freeness vs. SRE for HC and LC refining (80 kwh/t) for industrial pulp at Kappa 70 and 85**

### 4.3.2 Tensile strength

It was observed that for all HC refined samples, tensile index increased as SRE increased (Figure 25). An increase in SRE increased the level of refining which makes the fibres more collapsible. Fibres pack very closely together and strong bonds are created between them (Ebling, 1980; Hell, 1998). Increased interfibre bonding improves tensile index (Helle, 1998). The highest tensile index was reached after HC refining at 200 kWh/t for pulp at Kappa 70 (56 kNm/kg) and at 300 kWh/t for Kappa 85 (52 kNm/kg).

At the same SRE level (200 kWh/t), tensile strength was higher by 7 units for pulp at Kappa 70 compared to pulp at Kappa 85. At higher Kappa number, the fibres become stiffer and thus the fibre does not conform or consolidate well and as a result the bonds created between the fibres are weak (Alexander *et al* ., 1968; Jones, 1972). Tensile index was further improved after the LC refining stage. The reason for this is that LC refining stage is intended to straighten the fibre resulting in improved bonding ability (Gullichsen *et al.*, 1999). These results are summarized in Figures 26 and 27. The percentage of curled and kinked fibres was lower after LC refining. The values of tensile index for Kappa number 70 were higher by 8 units when compared to pulp at Kappa 85 at the same level of SRE.

The increase of tensile index after LC refining was significantly higher for pulp at Kappa 70 ($p=0.001$). This could be explained by severe fibre cutting of high Kappa pulp during LC refining due to the fibre being stiff (Figure 29).

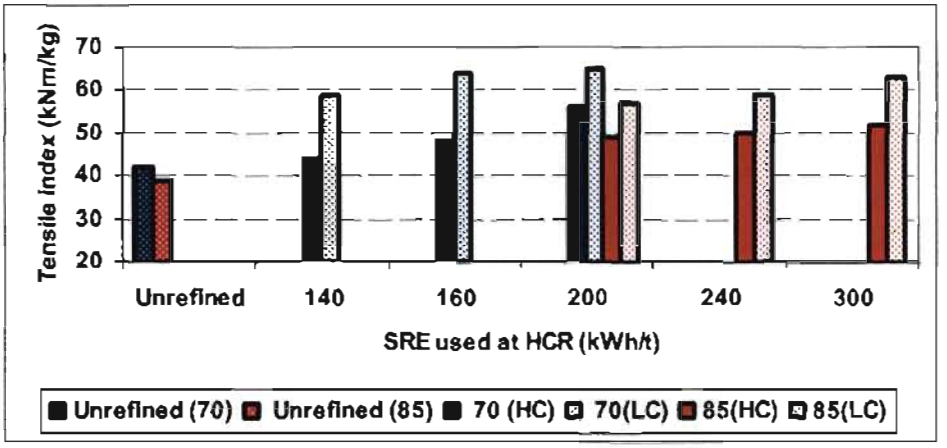



**Figure 25: Tensile index vs. SRE for HC and LC (80 kWh/t) for pulp at Kappa 70 and 85**

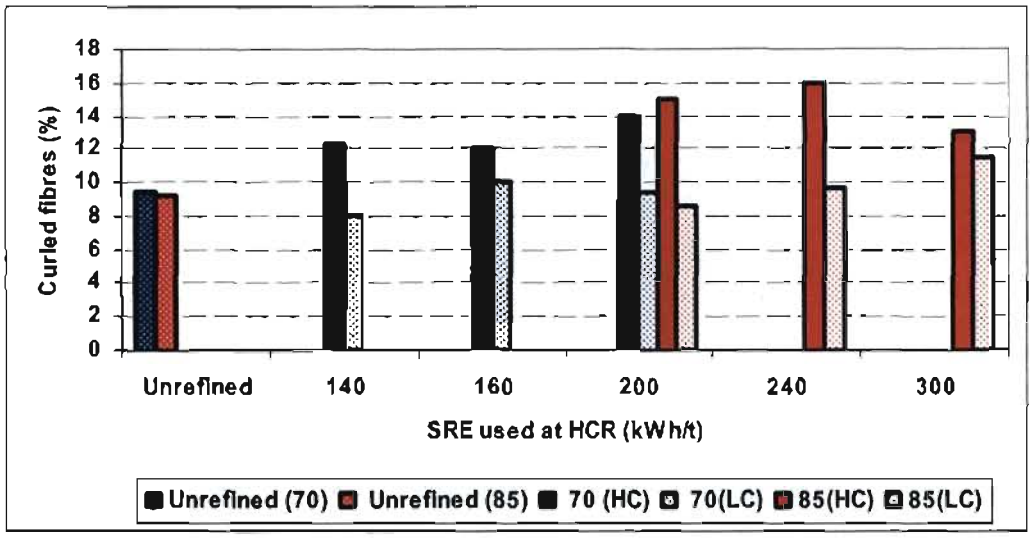



**Figure 26: Percentage of curled fibre vs. SRE for HC and LC (80 kWh/t) for pulp at Kappa 70 and 85**

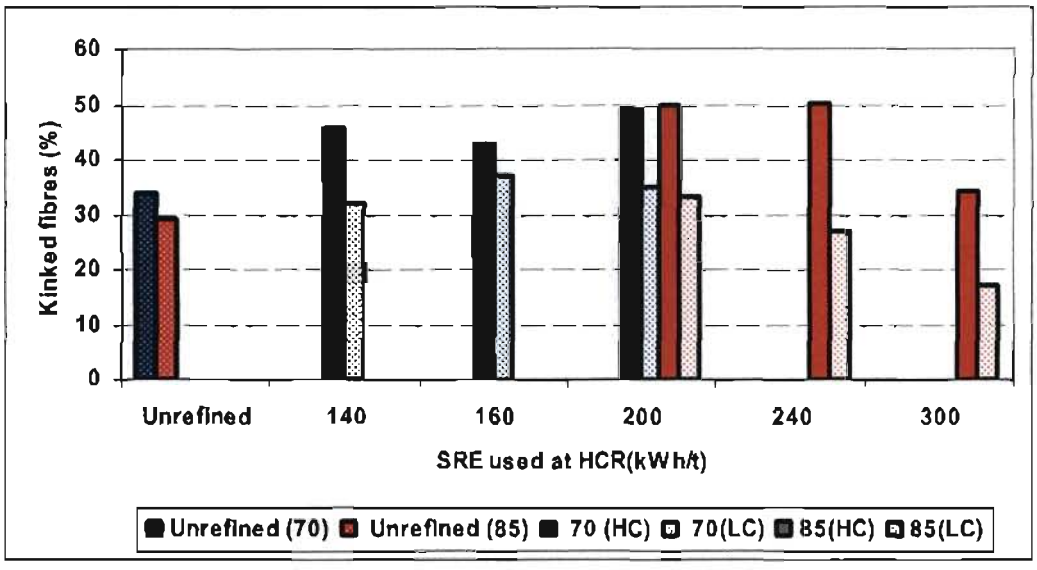



**Figure 27: Percentage of kinked fibres vs. SRE for HC and LC (80kWh/t) for pulp at Kappa 70 and 85**

### 4.3.3 Tear strength

HC and LC refining negatively affected tear index for both pulps (Figure 28). An increase in SRE caused a decrease in tear index. After HC refining, pulp at Kappa 85 had a higher tear index. This could be explained by the fact that at higher Kappa number the fibres are stiffer. Thick and stiff fibres have high tear resistance compared to thin and flexible fibres (Jones, 1972; Helle, 1998; Gullichsen *et al.*, 1999).

It can be seen that tear index was further reduced after the LC refining stage (Figure 28). This is because, although LC refining improves bonding ability, it also causes fibre cutting (Gullichsen *et al.*, 1999). Fibre cutting is most likely to be more severe for stiff fibres (Figure 29). After LC refining, pulp at Kappa 70 had a significantly higher tear index compared to pulp at Kappa 85.

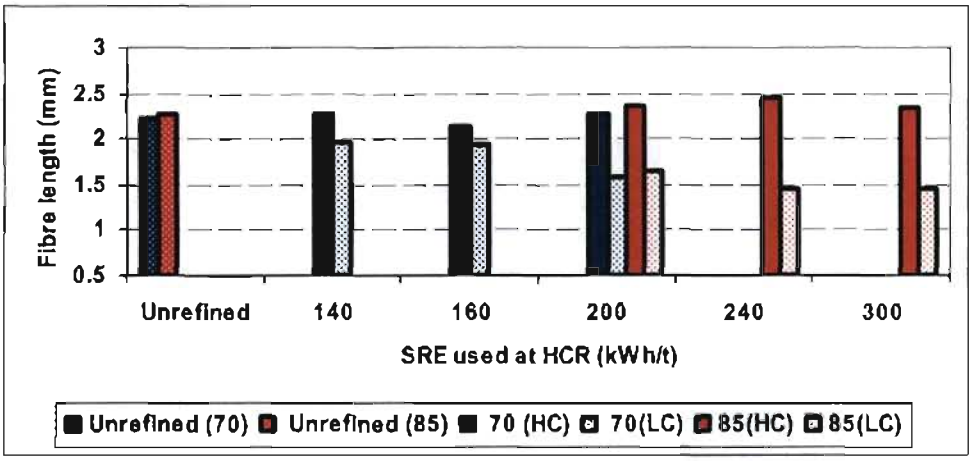



**Figure 28: Fibre length vs. SRE for HC and LC refining (80 kWh/t) for pulp at Kappa 70 and 85**

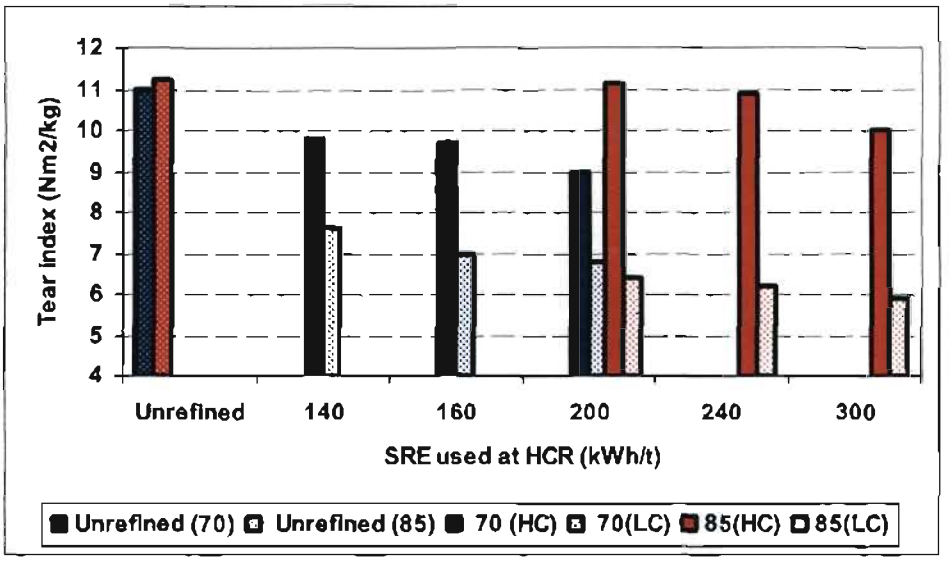



**Figure 29: Tear index vs. SRE for HC and LC (80 kWh/t) for pulp at Kappa 70 and 85**

### 4.3.4 Burst strength

After HC refining, pulp at Kappa number 70 reached a higher burst index at 200 kWh/t SRE. Pulp at Kappa 85 reached higher value of burst at 300 kWh/t SRE (Figure 30). However, the difference between the two pulps was not significant (p=0.6). After the LC refining stage, both pulps showed further improvement in burst index. However, the increment was significantly higher for pulp at Kappa 70 compared to pulp at Kappa 85 (Figure 30).

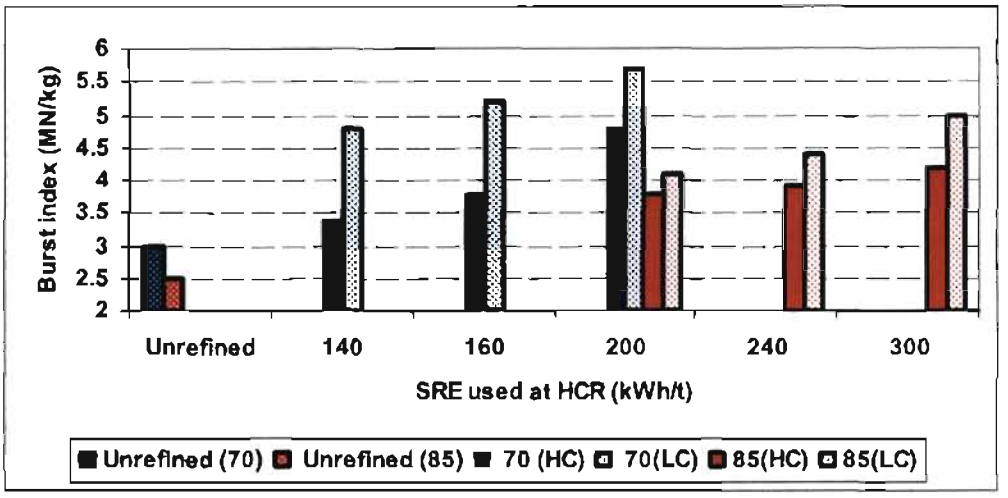



**Figure 30: Burst index vs. SRE for HC refining and LC refining (80 kWh/t) for pulps at Kappa 70 and 85**

### 4.3.5 Tensile energy absorption (TEA)

Figure 31 shows the relationship between tensile energy absorption (TEA) and SRE for the two pulps for HC and LC refining. After HC refining, pulp at Kappa 70 exhibited higher development in TEA index compared to pulp at Kappa 85. At the same SRE level, pulp at Kappa 70 had 53% higher TEA compared to pulp at Kappa 85. Further improvement of TEA occurred after LC refining for both pulps. It was found that TEA development was significantly higher for pulp at Kappa 70 after HC and LC refining compared to pulp at Kappa 85 ($p < 0.05$).

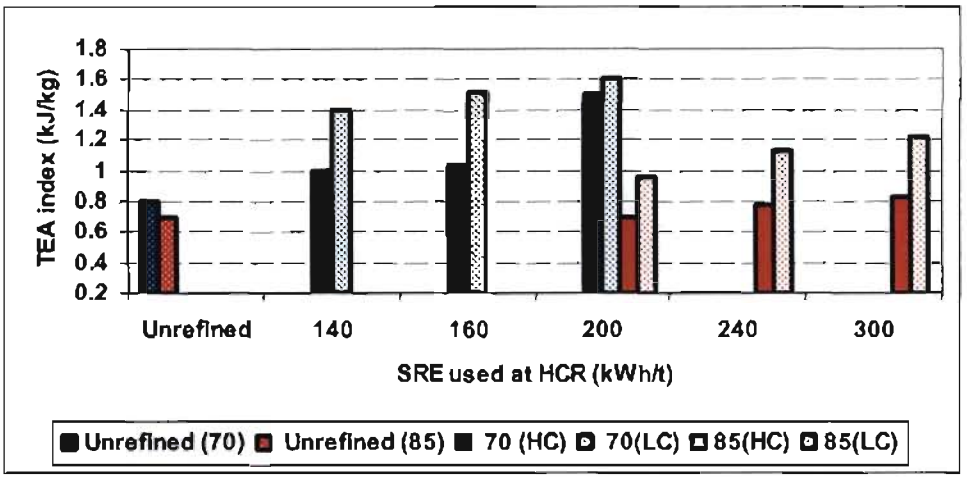



**Figure 31: TEA index vs. SRE for HC refining and further refining at LC (80 kWh/t) for pulp at Kappa 70 and 85**

The difference in TEA development between the two pulps could be attributed to degree of extensibility. Kappa 70 developed more stretch compared to pulp at higher Kappa number (Figure 32). High stretch enhances TEA (Pagaliarini, 1992; Gurnagul *et al.*, 2005).

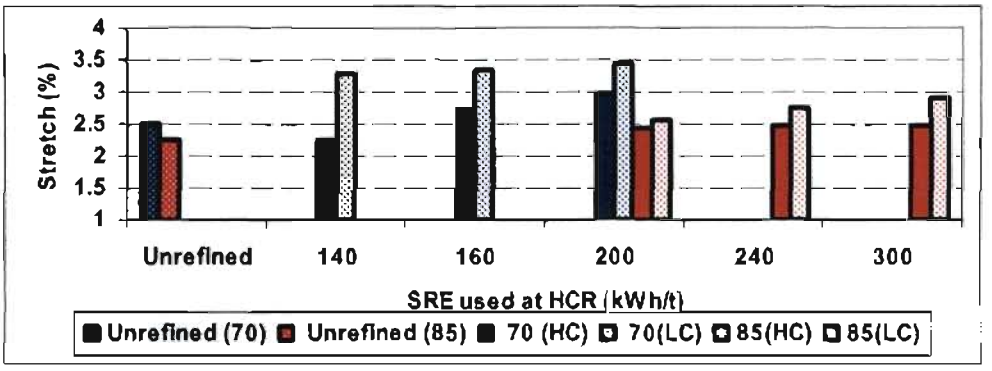



**Figure 32: Stretch for HC refining and LC refining (80 kwh/t) for pulp at Kappa 70 and 85**

### 4.3.6 Porosity

Airflow resistance is influenced by the level of refining and an increase in airflow resistance indicates reduced porosity (Hell, 1998; Gurnagul *et al.*, 2005). An increase in refining energy makes fibres more flexible, thus the fibres pack very closely together thereby reducing the number and size of pores in the sheet. As a consequence, airflow resistance increases as it becomes more difficult for air to escape. As a result, porosity is lower.

The results indicated that after HC refining the pulp was more porous compared to after LC for both pulps (Figure 33). This could be due to production of curl on fibres during HC refining which creates more voids between the bonded fibres (Gullichsen *et al.*, 1999; Gurnagul *et al.*, 2005). Pulp at Kappa number 85 had lower air flow resistance compared to pulp at Kappa 70 after HC refining probably due to stiffer fibres at Kappa 85. Stiffer fibres will not conform well and porosity will be higher.

After the LC refining stage, airflow resistance increased (Figure 33). This is because LC refining improves fibre collapsibility and bonding which reduces the number and size of pores in the sheet, thus increasing airflow resistance (lower porosity). There was a sharp increase in airflow resistance for Kappa number 85. This may be due to fibre cutting which resulted in fines generation. Fines filled the pores and voids in the sheet, resulting in an increase in air flow resistance (reduced porosity). It was observed that the difference in porosity between HC and LC refining was statistically significant for both Kappa numbers ($p < 0.05$).

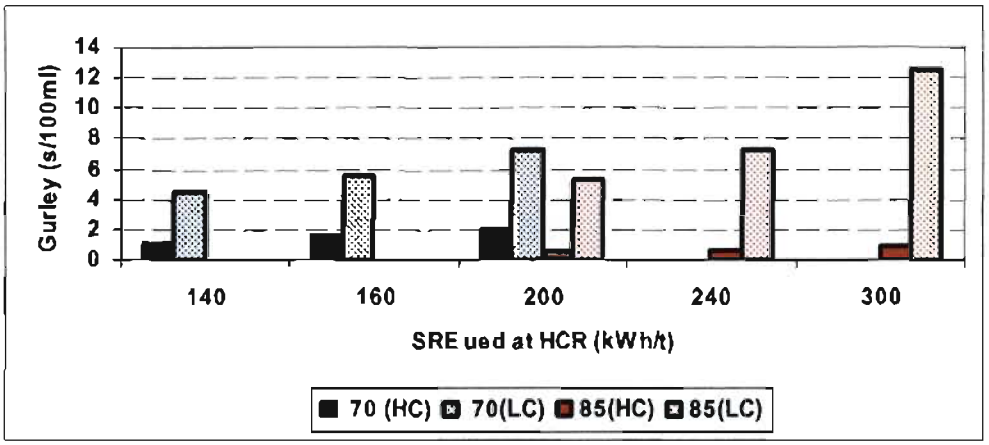



**Figure 33: Gurley vs. SRE for HC refining and further refining at LC (80 kWh/t) for pulp at Kappa 70 and 85**

### 4.3.7 Bending stiffness

After HC refining, bending stiffness decreased as SRE increased (Figure 34). This is because increasing SRE increases level of refining which makes the fibre more flexible. Flexible fibres have less resistance to bending (Hell, 1998). Pulp at Kappa number 85 had higher bending resistance (39%) compared to pulp at Kappa 70 at same SRE.

After LC treatment, bending stiffness was further reduced (Figure 34). However, pulp at kappa 70 (200 kWh/t) showed similar bending stiffness to pulp at Kappa 85 (300 kWh/t).

These results may suggest that Kappa 85 pulp can achieve similar bending stiffness as Kappa 70 provided sufficient refining is done (Figure 34). It was observed that for the level of SRE used, the difference in bending stiffness between the two pulps was not statistically significant for both HC and LC refining ($p > 0.05$).

It has been demonstrated in several studies that excessive resistance to bending (rigidity) causes the paper sheet to fracture, crepe or buckle (Hell, 1998). If this happens the paper sheet looses its original shape due to the deformation of the fibres in the sheet. Thus the more flexibility of the fibres in the sheet, the less the chance of sheet to buckle or crepe. This effect affects printing and sack papers in particular. Printing paper with crepe will stack in the printer or fax machines while creped sack papers will cause stacking problems in the conversion plant (Hell, 1998).

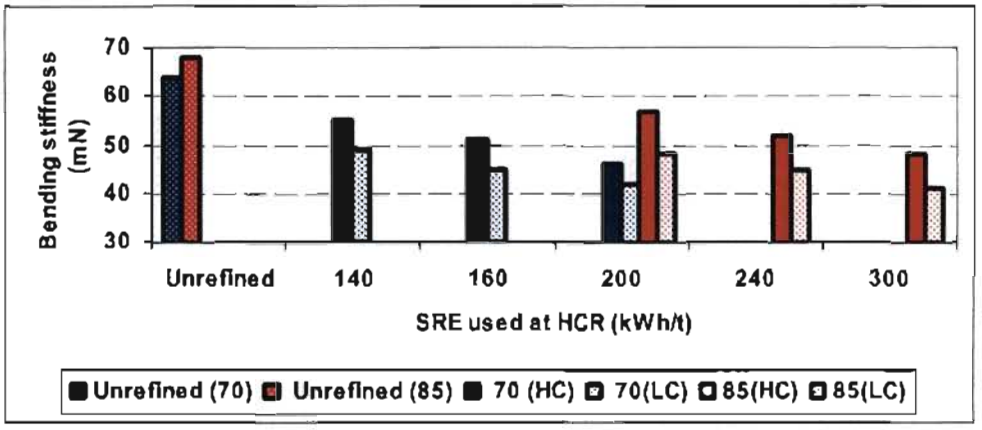



**Figure 34: Bending stiffness vs. SRE for HC refining and further refining at LC (80 kWh/t) for pulp at Kappa 70 and 85**

### 4.3.8 Zero span tensile strength

Zero span tensile strength is a measure of individual fibre strength (Helle, 1998; Andersson, 1999). It was observed that zero span tensile strength increased as SRE increased after HC refining (Figure 35). Kappa number 85 showed a slight lower value compared to Kappa number 70. However, the difference between the two pulps was not significant ($p=0.1$). After LC refining, zero span tensile strength was further improved (Figure 35). The differences in the magnitude of zero span tensile development between the two pulps was found to be significant ($P=0.02$).

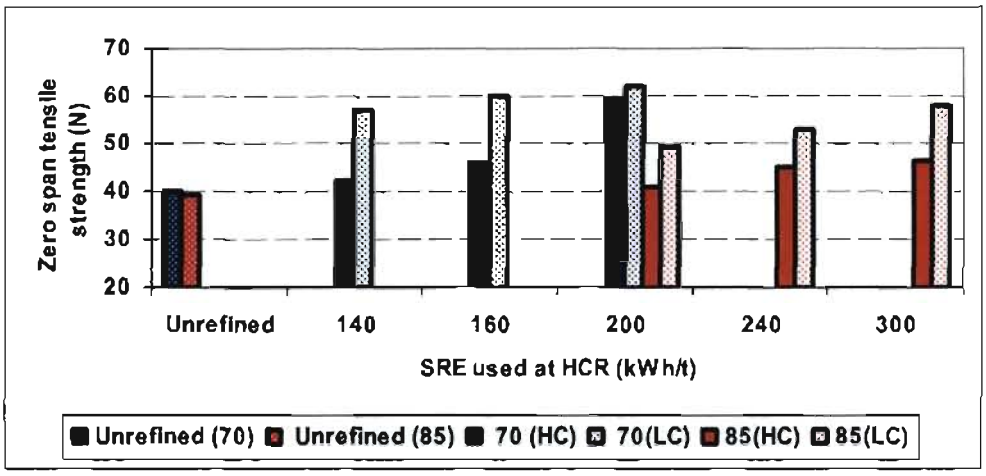



**Figure 35: Zero span tensile strength vs. SRE after HC and LC refining (80kWh/t) for pulp at Kappa 70 and 85**

## 4.4 Conclusions

The objective of this study was to understand the impact of HC and LC refining on strength property development for high Kappa number pulps. The influence of Kappa number and SRE on strength of industrial pulps was studied. The results showed SRE level for optimum strength development depended on the Kappa number of the pulp. For instance most of the optimum strength properties for pulp at Kappa number 70 were obtained at 200 kWh/t and at 300 kWh/t for pulp at Kappa number 85.

However, pulp at Kappa 85 showed inferior tear index, TEA, stretch and a rapid drop in porosity. This may have a negative impact on commercial pulp, particularly if the pulp is intended for sack paper grade. The rapid drop in porosity may have effect on paper machine speed as well as web breakage. Based on these results there is no advantage to be gained by using pulp at Kappa 85 for sack grade Kraft.

The evaluation on energy input per unit Kappa number showed that an average increase of 15 units of Kappa number requires an average increase of 100 units as total energy input (HC and LC combined).

# CHAPTER 5: POTENTIAL OF DEVELOPING HIGH PULP STRENGTH FROM HIGH YIELD KRAFT PULP MADE FROM *PINUS PATULA*

## 5.1 Introduction

The focus of this component of the work was to provide a better understanding of whether the yield gain and the level of strength properties attained will provide an economic benefit for HYK pulp production, in particular sack paper grade. In addition, the information obtained in this study could be used by the paper industry either to adjust pulping and refining conditions or defining the most appropriate application of high Kappa number Kraft pulps.

## 5.2 Material and methods

### 5.2.1 Field sampling

*Pinus patula* was sampled from Demagteburg plantation in a compartment E1 situated in Karkloof (Sappi forest) located in KwaZulu-Natal Midrands. Twelve trees were randomly selected and felled. Seventeen kilograms of wood chips were required for each refining trials. Two meter logs were sampled from each tree. Table 9 shows information on the fibre source.

**Table 9: Information of the fibre source**

| **Wood species** | ***Pinus patula*** |
|---|---|
| Age (years) | 12 |
| Reference age (years) | 20 |
| Site index | 23 |
| Mean total height (m) | 13.6 |
| Standard deviation(SD) | 0.45 |
| Mean DBH (cm) | 17 |
| Standard deviation (SD) | 1.74 |
| Basic density ($gcm^{-3}$) | 0.36 |
| Standard deviation (SD) | 0.05 |

### 5.2.2 Experimental design

Chips were cooked using the conventional Kraft pulping method. The pulping conditions are detailed in Figure 36. After pulping, chips were defibrated to separate fibre bundles. Pulp was then screened in a wire mesh screen (1.7 mm). Coarse rejects were recovered and defibrated (Figure 36). Pulps were then dispersed in a mixer to remove latency followed by screening. A sample of screened pulp was used to evaluated pulp pH, Kappa number and shive content. Pulps at Kappa 100, 115 and 130 were produced and refined using the HC and LC refiners. The conditions used for the refining and the list of pulp tests are detailed in Figure 37. Detailed explanations of pulping and refining procedures are described in section 5.2.3.

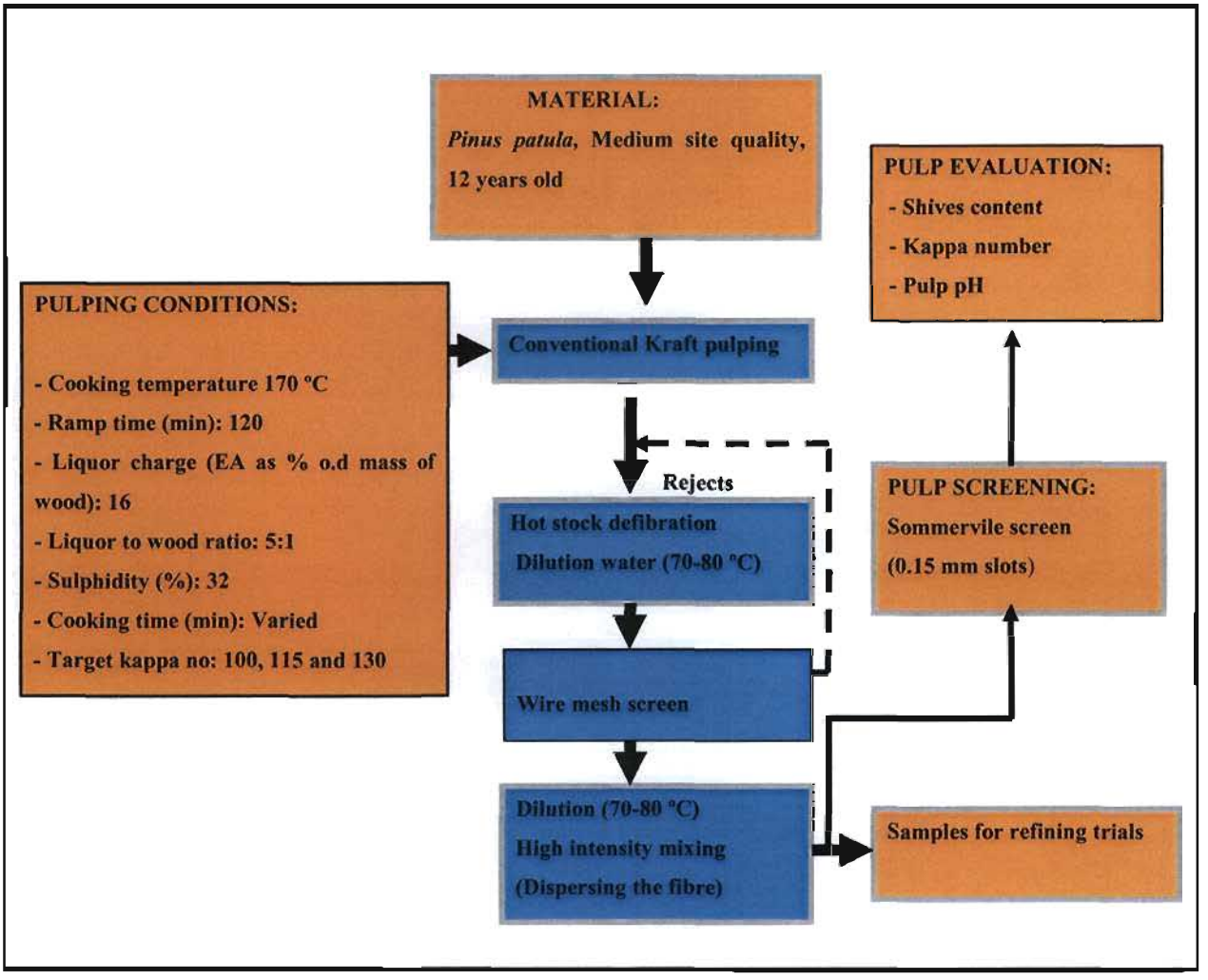



**Figure 36: Experimental design for pulping trial**

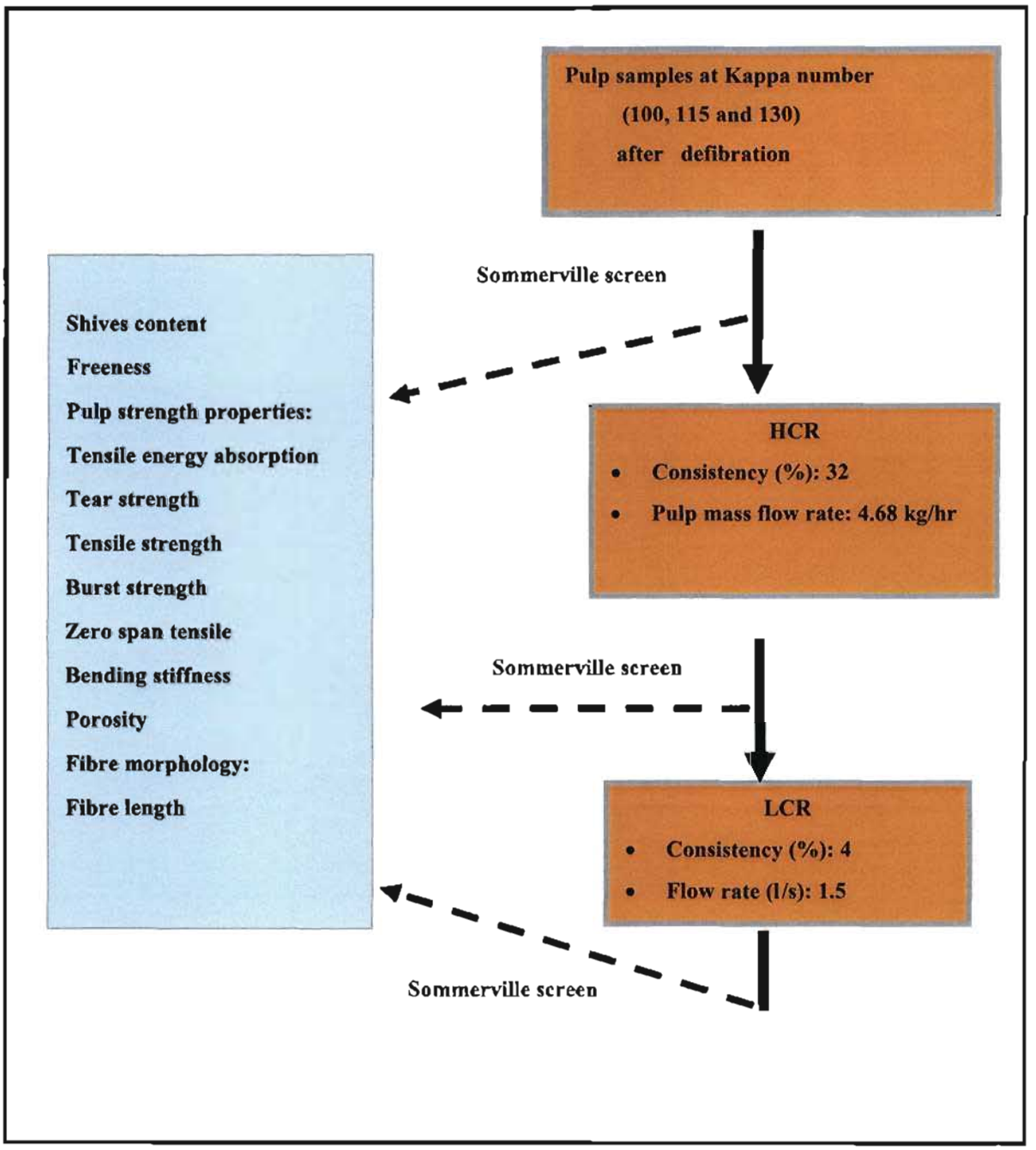



**Figure 37: Experimental design for refining trials**

### 5.2.3 Experimental procedure

- **Sample preparation**

The logs were debarked using a hand axe. The debarked logs were chipped using a wood laboratory chipper (precision Husky type). The chipper was provided with a knife gauge. The chipper knife gauge could be adjusted to various knife widths depending on chip size required.

In this study, the chipper knife gauge was set at ¾" knife width (chipper size 38") which produces woodchips with an average length of 15-30 mm. The average woodchip thickness falls in the range of three to six millimetres. This setting of the chipper was used for producing all of the wood chips used in this project. The chips were screened using a vibrating screen to remove both undersize and oversized chips which has a detrimental effect on the pulping process (Christensen, 1998). Non-uniform size wood chips affect pulp uniformity and also may lead to under utilization of the digester capacity. Pin chips and fines (undersize) have been reported to hinder liquor circulation in the digester (Christensen, 1998).

The chips were allowed to air dry for two weeks to allow wood chip moisture content to reach equilibrium (Christensen, 1998). Woodchip moisture content was determined according to TAPPI test method T258om-94 (Kocurek, 1992, TAPPI test method, 2000). Wood moisture content is important in determining the amount of water and black liquor to be added to make up the liquor to wood ratio required (Kocurek, 1992; Christensen, 1998). The chips were then stored in a sealed plastic bag to ensure the woodchip moisture content was not affected by the atmospheric or weather changes.

- **Preparation of cooking liquor**

Cooking liquor was prepared and standardized according to TAPPI test method T624 cm-85 (Kocurek, 1992, TAPPI test method, 2000). The cooking liquor specifications were total alkalinity of 168.6 g/ll as $Na_2O$; Effective alkali of 156.9 g/ll as $Na_2O$ and sulphidity of 32%. The quality of the liquor in terms of effective alkali and sulphidity were measured regularly and standardized as shown in appendix A-4. This was to check whether the liquor has been significantly oxidized or not. Oxidation of the cooking liquor results in a decrease in effective alkali as well as sulphidity which consequently affects the pulping results (Kocurek, 1992; Christensen, 1998).

- **Pulping**

All trials were conducted using a laboratory flow- through digester with a capacity of 300 l. The digester was manufactured by Metso ND Engineering (Pty) Ltd to handle projects of this nature. The target Kappa numbers were 100, 115 and 130. The flow- through digester is equipped with three batches of heaters (top, middle, and bottom) for indirect heating. It has a cooking liquor circulation and temperature control system. These features enhance uniform heat and cooking chemical distribution throughout the entire digester (Christensen, 1998; Van Tran, 2005).

After screening, only wood chips with an average chip thickness of three to six millimetres were pulped. The mass of wood chips charged was 17 kg (14.28 kg oven dry mass) while

cooking liquor charge was 16% (as effective alkali on oven dry mass). To ensure that all the woodchips in the digester were submerged in liquid, a liquor to wood ratio of 5:1 was used. To enhance cooking liquor impregnation prior to the start of pulping, the digester was pressurized for 20 minutes using the liquor circulation pump until the temperature reached 75 °C. This was aimed at minimizing the localization of cooking liquor components as well as the variation of temperature gradient in the digester (Kocurek, 1992). This procedure also simulated the industrial pulping to some degree, where wood chips normally enter into digester at about 75-80 °C (Kocurek, 1992).

The ramp-up phase was from 75 °C to 170 °C at a ramping rate of 1 °C/minute (all three heaters were used). The temperature was held at 170 °C for the desired cooking time/ H-factor. This was done by switching the heaters (1 and 2) on and off, as well as the inlet cooling water valve. At the end of each cook all the heaters were switched off. In order to enhance cooling, cooking liquor was left to circulate through the pump for the entire cooling period. The cooling phase was terminated at 40 °C. The cooling rate was found to be about -2.3 °C/min. The digester cooling process was aimed to reduce the effect of blowing the digester at high pressure (temperature). Blowing the digester at high temperature has been reported to have a negative effect on pulp strength (Christensen, 1998). In mills, this is achieved by the use of a relief valve to reduce the digester pressure prior to blowing (Christensen, 1998).

At the end of each cooling phase, the drain valve was activated and a sample of black liquor was collected for analysis. The black liquor analysis results are illustrated in appendix A-4 (Table A4-3). It was found that the residual alkali after a cook was in range of six to seven gram per litre and was not significantly different compared to that quoted in the literature for a pulp yield of less than 70% (Kocurek, 1992). This was followed by pre-washing of the mildly cooked chips.

The washing process was accomplished by following the black liquor displacement principle. This was done by filling the digester with hot water using a hose pipe, while at the same time draining the black liquor out. This was followed by completely filling the digester with hot water and circulating it with the pump while draining the cooking liquor out, until the drain water was almost colourless. Figure 38 shows the process flow diagram of the laboratory flow-through digester used for the pulping experiments. The digester temperature was calibrated prior to the start of the trials and the results are graphically shown in Appendix A-3. Pulping temperature varied within the range 170±3 °C.

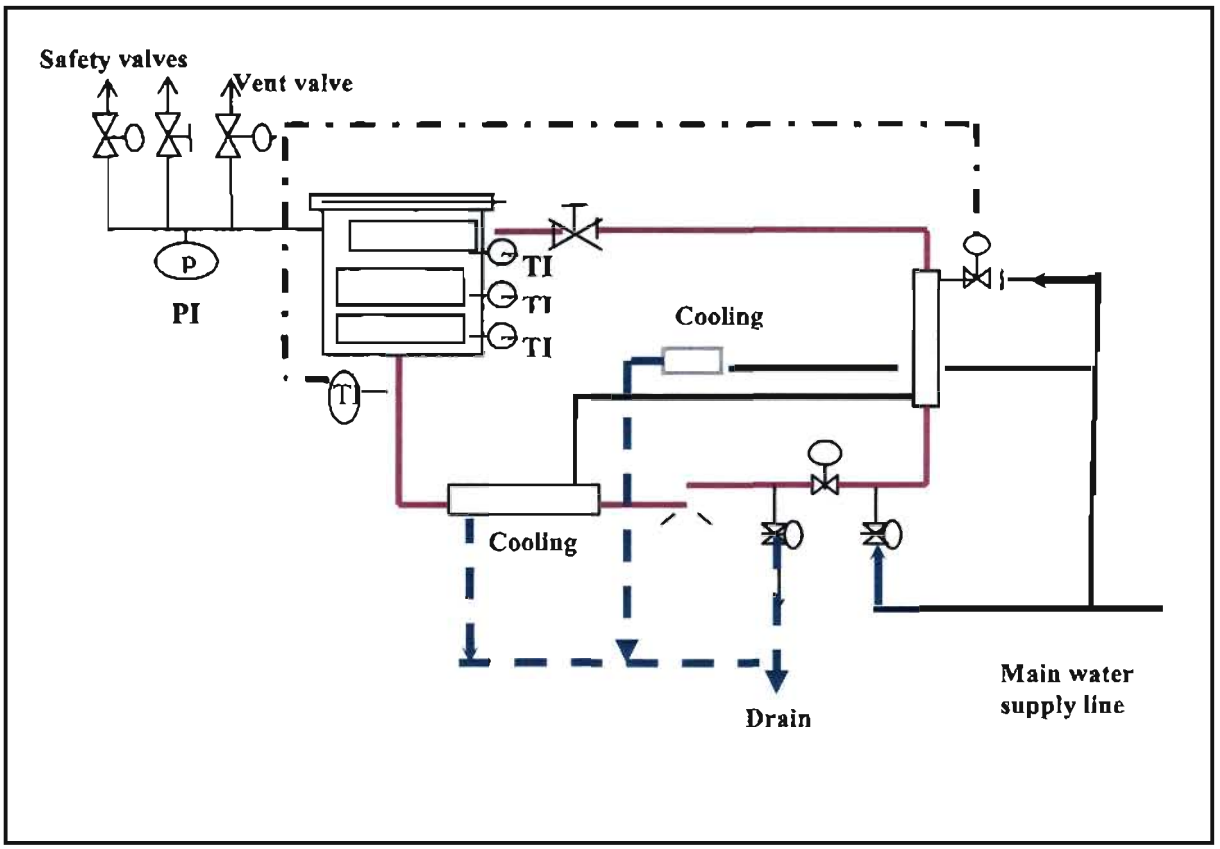



**Figure 38: Process flow diagram of the laboratory flow through digester**

- **Defibration and pulp screening**

It is well accepted that fibre liberation in conventional Kraft pulping is limited at a yield of 50% (McGovern, 1972; Kurdin, 1981). However, practically, it depends on the pulping uniformity. To separate and to eliminate the fibre bundles, defibration is carried out in the refiner with defiberizing discs. Thus defibration was necessary in order to separate the fibre bundles (Hartler *et al.*, 1976; Andersson, 1999; Floris, 2000).

All the defibration trials were done using the high consistency refiner equipped with defibration plates. These plates are designed to separate fibres and not to refine (source: IPT South Africa). All the mildly cooked chips were first slashed by using a gap clearance of 750 microns for one pass. This was aimed to reduce the size of the mildly cooked chips, and then all the samples were defibrated by passing through a gap clearance of 375 micron for two passes. To enhance the fibre separation effect with minimum fines generation, hot stock defibration was adopted (Hartler *et al.*, 1976).

Primary screening was done using a wire mesh screen (1.7 mm), and the rejects were collected and recycled back to the process (Figure 35). Each pulp sample was diluted with

warm water (80-100 °C) in the refiner chest tank and the pulp suspension was agitated for 30 minutes. This was aimed at minimizing latency which could affect the efficiency of screening (Helle, 1998; Jarkko, 2005). Then each pulp sample was spin dried for 20 minutes. All of the pulp samples were weighed and packed in plastic bags and stored in a fridge at 4 °C. For each pulp sample, 2000 g was taken for shive content determination, Kappa number analysis as well as handsheet making. Shives were removed from the pulp sample for handsheet making, because shives initiate micro fracture in the sheets resulting in a negative effect on the pulp strength properties (Helle, 1998).

- **Refining**

SRE applied to the three pulps at different Kappa numbers are detailed in Table 10. This energy input estimate was based on results obtained in Chapter four which indicated that total energy input (HC+ LC) would need to be increased by 100 kWh/t for every 15 unit increase in Kappa number.

**Table 10: Energy input used in various refining stages for each Kappa number**

| Kappa number | SRE (kWh/t) for HCR | SRE (kWh/t) for LCR | Total SRE (HC+LC) kWh/t |
|---|---|---|---|
| 100 (K100) | 300; 400 | 80; 120 | 380; 420; 480; 520 |
| 115 (K115) | 400; 500 | 120; 180 | 520; 580; 620; 680 |
| 130 (K130) | 500; 600 | 180; 200 | 680; 700; 780; 800 |

- **Pulp evaluation**

The moisture content was determined according to TAPPI test method T258om-94 (Kocurek, 1992, TAPPI test method, 2000). The total yield for each pulp sample was evaluated as a percentage of mass of oven dried defibrated pulp to mass of oven dried wood chips charged into digester. The degree of delignification was determined using the Kappa number test according to TAPPI test method T236cm-85 (TAPPI test method, 2000; Kubes *et al.*, 2002). The samples used for determination of Kappa number were washed and then screened using a sommerville screen with 0.15 mm slots to remove shives, before testing. Pulp with high traces of black liquor as well as shives affects Kappa test results (Christensen, 1998).

- **Shive content**

For each pulp sample, 2000g was taken for determination of shive content in the pulp mass. The pulp samples were screened in the sommerville screen with 0.15 mm slots (Hartler *et al.*, 1976; Xu *et al.*, 2002). The shives which were retained on the top of plates were collected and dried in the oven. The mass of shives was expressed as percentage of the pulp sample mass on an oven dry basis. The amount of shives after defibration was determined in order to assess the effect of HC and LC refining on shive reduction in the pulp mass.

- **Pulp pH**

The pulp pH was determined according to modified TAPPI test method T252-0M-90 (Kocurek, 1992, TAPPI test method, 2000). 10 g of pulp was diluted with 800 ml of deionized water. The suspension was stirred for five minutes. The pH meter probe was then inserted in the pulp suspension and the reading was recorded for each pulp sample (pulp pH = p $H_{Final}$ – $pH_{H2O}$). The pulp pH was used to measure the effectiveness of pulp washing. In addition to that pulp pH has an effect on fibre strength development during HC refining (Helle, 1998). Thus the pulp pH should be close to 8. The pH values are shown in Appendix A-4. The experimental pH values were not significantly different to the recommended value, and thus the effect of pulp pH during HC was considered negligible.

- **Freeness**

After refining, the Canadian Standard Freeness (CSF) measurement was performed according to TAPPI test method T227 om-94 (TAPPI test method, 2000, Xu and Sabourin, 2002).

- **Preparation of hand sheets and testing of sheet physical properties**

The same methods used in Chapter three were used for this study.

- **Fibre morphology**

The same methods used in Chapter three were used for this study.

## 5.3 Results and discussion

### 5.3.1 Pulping results

- **Influence of H-factor on the rate of delignification**

As was anticipated, an increase in H-Factor resulted in a decrease in Kappa number as shown in Figure 39. It was observed that an increase of 154 units of H-factor corresponded to a

reduction of 25 units in Kappa number. A good correlation was found between H-factor and Kappa number ($R^2$=0.999). The results suggested that under well-controlled pulping conditions, H-factor can be used to predict Kappa number as it has been reported in previous studies (Vroom, 1957; Kleppe, 1970; Christensen, 1998; Kubes *et al.*, 2002).

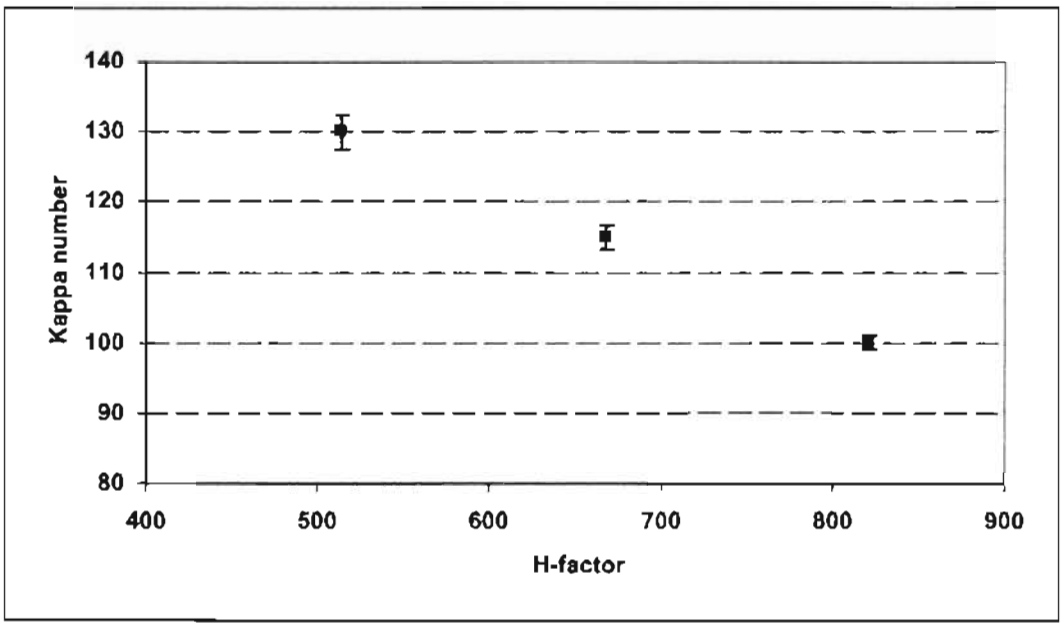



**Figure 39: Graph of H-factor vs. Kappa number**

- **Influence of Kappa number increase on total pulp yield**

An increase in Kappa number corresponded to an increase in total pulp yield (Figure 40). It was observed that an increase of an average of 15 units of Kappa number corresponded to an increase of 1.6% in total pulp yield. The results showed a good correlation exists between Kappa number and total pulp yield ($R^2$=0.9685). In a similar study on the range of Kappa number (100-150) based on Northern pine (Kleppe, 1970; Smoke, 1992), the average yield increase was 1.8%.

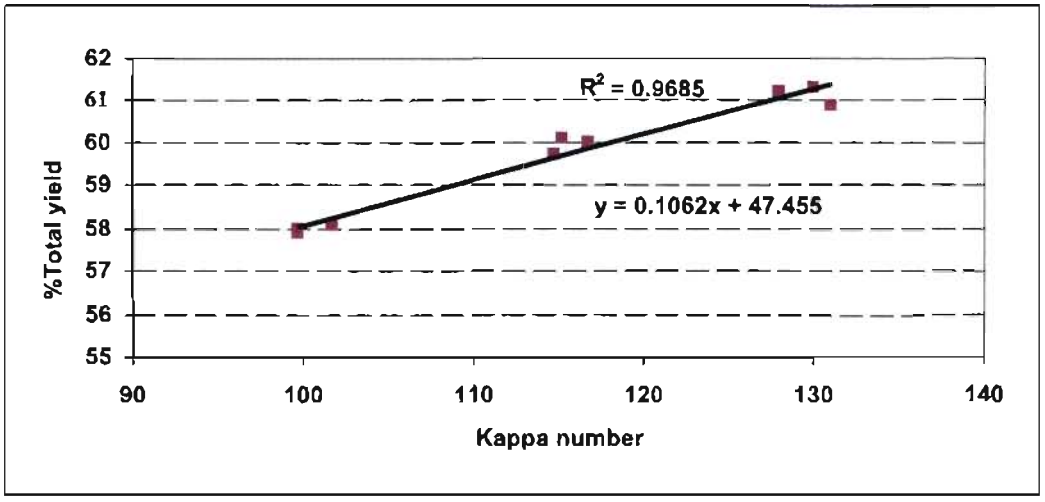



**Figure 40: Graph of Kappa number vs. total yield**

- **Influence of Kappa number on pulping uniformity**

An increase in Kappa number resulted in an increase in shives in pulp (Figure 41). The percentage of shives was 10, 28, 44, and 47 at Kappa 70, 100, 115 and 130 respectively. Pulping to high Kappa number by conventional method affects pulping uniformity because the cooking time is short. As a result the degree of liquor penetration into the wood chip is limited (diffusion-limited), and consequently some of the wood mass is not effectively exposed to the cooking liquor resulting in under- cooked fibre bundles. Probably the use of small wood chips size could improve liquor impregnation, thus improve the pulping uniformity (McGovern, 1972).

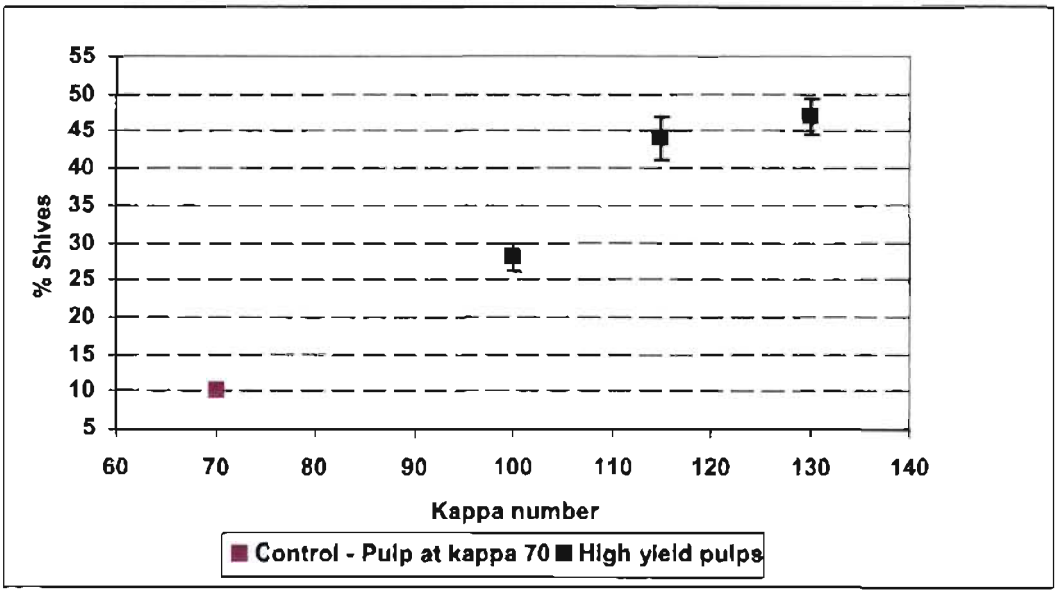



**Figure 41: Graph of Kappa number vs. Shives**

### 5.3.2 Refining results

- **Influence of HC and LC refining on shive reduction**

The amounts of shives expressed as a percentage for each pulp sample are illustrated in Figure 42. It was found that after the HC refining stage the pulp at each Kappa still contained high shive content. However, after the LC refining stage the shives were significantly reduced (p=0.001). The results suggest that in order to reduce the shive content in high yield pulp, LC refining is an essential stage. Practically, in industry, pressurized secondary refining is used for all low shive pulp grades (Xu and Sabourin, 2002).

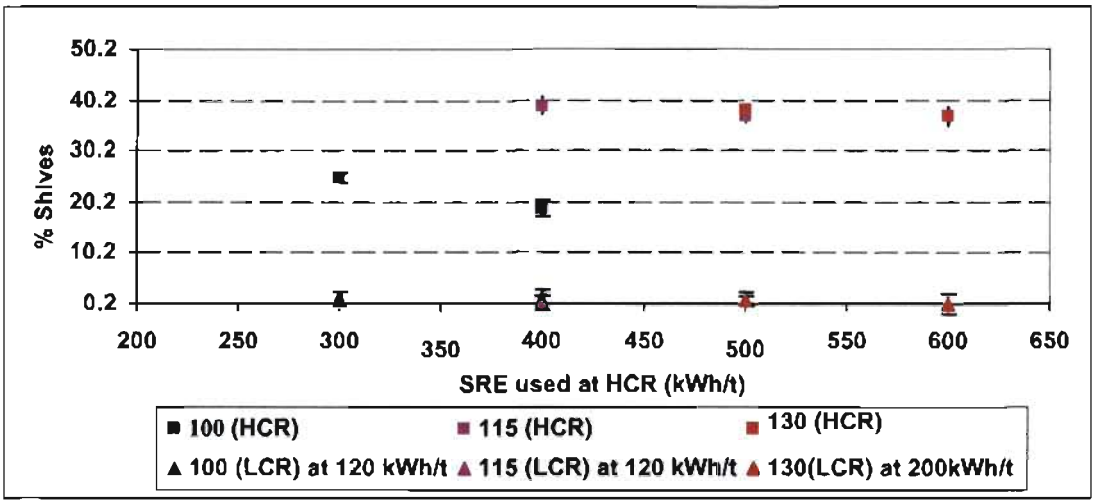



**Figure 42: Graph of shive content vs. SRE for pulp at different Kappa numbers**

- **Effect of refining on Kappa number reduction**

In order to understand the effect of refining on lignin dissolution, the Kappa number of whole pulp from each refining stage (HCR and LCR) were evaluated and compared with the Kappa number of shives after HCR for each pulp sample. The results showed that refining resulted to some degree in lignin dissolution, as the Kappa numbers were reduced (Table 11). The Kappa number reduction is due to the breakage of fibre cell walls which exposes the lignin, and the lignin is then leached from the fibres (Lindstrom *et al.*, 1978; Li and Macleod, 1992; Andersson , 1999).

However, lignin dissolution was more effective at higher Kappa (Table 11). The comparison between blowline Kappa (after defibration) and final Kappa (after LC refining), shows a drop in Kappa number of 7, 8 and 10 units with respect to 100, 115 and 130 Kappa respectively. These results suggest that high Kappa number Kraft pulps have hard shives. Hard shives

break into smaller particles rather than being fibrillated, and lignin remains intact in the fibre cell wall. Such small fibre particles do not effectively play a part in bonding (Helle, 1998), consequently affecting the development of the strength properties, as is always the case for mechanical pulp.

**Table 11: Effect of refining on Kappa number reduction of high yield Kraft pulps**

| **Kappa number of Screened pulp** | **Kappa 100** | | **Kappa 115** | | **Kappa 130** | |
|---|---|---|---|---|---|---|
| *Refining stage* | *HCR* | *LCR* | *HCR* | *LCR* | *HCR* | *LCR* |
| SRE (kWh/t) used | 400 | 120 | 400 | 120 | 600 | 200 |
| Kappa of shives after HCR | 97 | - | 117 | - | 138 | - |
| Kappa of whole pulp (Including shives) | 95 | 93 | 100 | 97 | 123 | 120 |

- **Influence of Kappa number on pulp strength development**

The refining results were evaluated to identify the SRE for both HC and LC refining where the strength properties attained the highest values. These are given in Table 12 and graphically shown in Appendix B-4. Since most of pulp strength properties depend on freeness as well as hand sheet bulk (Hell, 1998; Xu and Sabourin, 2002), the relationship between freeness and hand sheet bulk was evaluated and graphically presented in Figure 43. All the samples exhibited high sheet bulk (1.2- 1.3 $cm^3/g$) for HC samples and 0.9-1.15 $cm^3/g$ for LC samples. More importantly it was found that there was no significant difference in sheet bulk after both HC and LC refining ($P>0.24$).

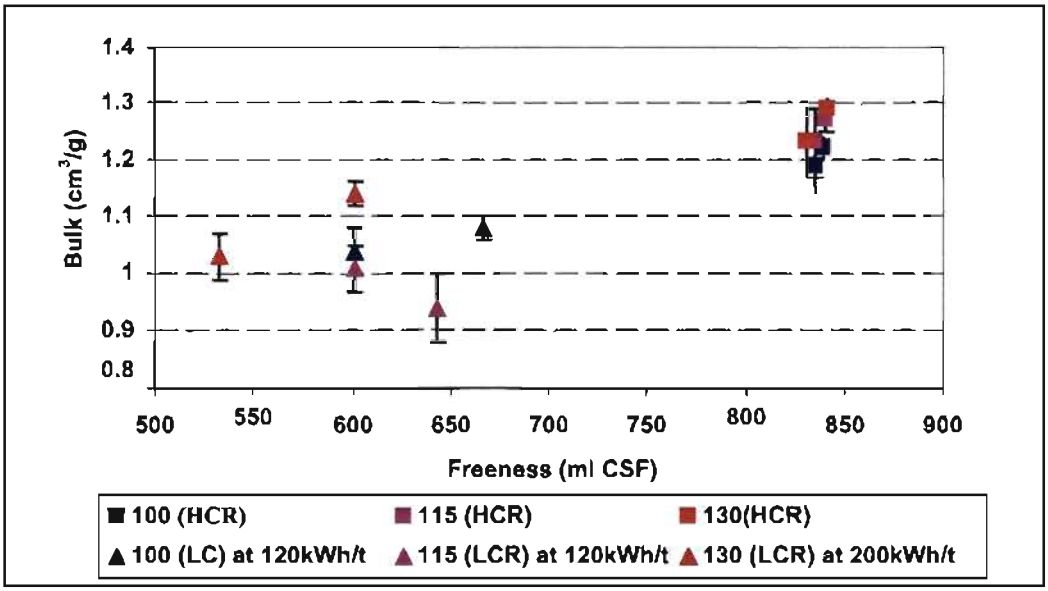



**Figure 43: Graph of freeness vs. SRE for HC and LC refining**

These results were supported by the values of strength properties (Table 12). It can be seen that high Kappa pulps have inferior strength properties compared to pulp at Kappa 70 with the exception of bending stiffness. Bending stiffness results indicated that pulp at Kappa number 100 can attain similar bending stiffness as pulp at Kappa 70 provided that sufficient refining is done (Table 12). The overall results suggest that at high Kappa number the degree of stiffness and brittleness is very high. Thus even a high level of refining creates only severe fibre cutting rather than increasing the fibre flexibility which is responsible for strength development as revealed by fibre length results. The fibre length for pulp at Kappa 70 was higher by 47%, 140% and 162% compared to fibre length for pulp at Kappa100, 115 and 130 respectively when referring to LC refined samples.

**Table 12: Potential of developing high strength from high yield Kraft pulp**

| Refining stage | Control Kappa 70 | | Kappa 100 | | Kappa 115 | | Kappa 130 | |
|---|---|---|---|---|---|---|---|---|
| | *HCR* | *LCR* | *HCR* | *LCR* | *HCR* | *LCR* | *HCR* | *LCR* |
| SRE (kWh/t) applied | 200 | 80 | 400 | 120 | 400 | 120 | 600 | 200 |
| Pulp properties | | | | | | | | |
| Freeness (ml CSF) | 668 | 575 | 835 | 601 | 840 | 643 | 831 | 532 |
| Tensile index (KNm/kg) | 52 | 66 | 34 | 40 | 33 | 37 | 25.5 | 27 |
| Tear index ($Nm^2/kg$) | 9 | 7.7 | 13 | 7.6 | 12.7 | 5.5 | 11 | 4 |
| Burst index (mN/kg) | 4.1 | 5.7 | 2.52 | 3 | 1.9 | 2.65 | 1.5 | 2.4 |
| TEA index (kJ/kg) | 1.32 | 1.6 | 0.61 | 0.7 | 0.43 | 0.62 | 0.31 | 0.33 |
| Stretch (%) | 3 | 3.4 | 2.45 | 2.65 | 2.1 | 2.37 | 1.8 | 1.9 |
| Porosity (Gurley s/100ml) | 2 | 7.2 | - | 1.4 | - | 3.4 | - | 2 |
| Bending stiffness (mN) | 46 | 42 | 49 | 41 | 53 | 36 | 48 | 34 |
| Zero span tensile strength (N) | 59 | 62 | 30 | 33 | 28 | 30 | 23 | 25 |
| Sheet bulk ($cm^3/g$) | 1.02 | 0.8 | 1.19 | 1.04 | 1.27 | 1.01 | 1.23 | 1.03 |
| Shives (%) | - | - | 19 | 1 | 39 | 0.5 | 37 | 0.24 |
| Fibre length (mm) | 2.28 | 1.94 | 2.12 | 1.32 | 1.75 | 0.81 | 1.73 | 0.74 |

- **Potential application of high Kappa number Kraft pulps**

The potential applications of HYK pulps are detailed in Table 13. It showed that the high Kappa number Kraft pulps can be useful for some other paper grades for which TEA is not critical quality requirement, such as fluting and Kraft liners. However, further investigation is required to characterize specifically the application of high Kappa numbers (115-130) for fluting or Kraft liners.

**Table 13: Pulp quality properties for various applications (Hell, 1998; Gurnagul *et al*, 2005; Sappi Tugela mill technical data)**

| Pulp properties | Commercial specifications | | Trial values |
|---|---|---|---|
| | *Other paper grade* | *Sack or linerboard* | |
| **Tensile index (kNm/kg) | 10-100 | 75 | 27-40 |
| **Burst index (mN/kg) | 1-10 | 1-10 | 2.4-3 |
| **Tear index ($Nm^2/kg$) | 6-30 | 6-23 | 4-7.6 |
| TEA index (KJ/kg) | 1-5 | 2-4.23 | 0.33-0.7 |
| * Stretch (%) | NA | 5-8 | 2-3 |
| * Porosity: Gurley (s/100ml) | NA | 6 | 1.4-3.4 |
| * Bending stiffness (mN) | NA | NA | 34-41 |

** Applicable to sack and linerboard and * Applicable to sack paper only

## 5.4 Conclusions

Pulping trial results indicated that pulping *Pinus patula* to a higher Kappa number offers a significant yield increase (58-61.5%) compared to yield at Kappa 70 which is limited at yield range of 42-48% (Kleppe, 1970; McGovern, 1972; Andersson, 1999; Tugela mills technical data). An average increase of 1.6% for every 15 unit increase in Kappa is comparable to 1.8% which is reported in the literature (Kleppe, 1970). However, reducing the liquor charge e.g from 16% to 10% (EA) and pulping at lower temperature or combination of these could further improve pulp yield.

However, the refining results indicated that improving pulp yield of *Pinus patula* through conventional Kraft pulping to Kappa number (> 70) had a negative effect on pulp strength properties for sack paper grade. These results agree with Chapter 4 results, where it is shown that industrial pulp at Kappa 85 from MCC digester showed inferior strength despite been refined at higher SRE. On the other hand, Kringstad and Vikstrom (1975) and Hartler *et al.* (1976) showed similar results. In addition, the introduction of process such as delignifying high Kappa number pulp to Kappa 70 also supports the finding in this study (Kleppe and Storebraten, 1985; Minja, 1998).

However, pulping at lower temperature may give better results. Pulping at lower temperature provides a gentle cook, thus enhancing pulping uniformity which is limited at high temperature due short cooking time. Pulp at Kappa (115-130) can be possibly utilized for other grades such as fluting and Kraft liners which TEA is not a critical quality requirement. The overall findings and recommendations are reported in Chapter six.

# CHAPTER 6: CONCLUSIONS AND RECOMMENDATIONS

## 6.1 Conclusions

The experimental results have provided sufficient understanding of the following areas:

- The pilot scale laboratory refiners (HC and LC) at the FFP laboratory can be used to simulate industrial refining processes.
- In optimizing fibre strength, both Kappa number and SRE need to be taken into consideration.
- The laboratory flow through digester has been built and is in operational to simulate conventional Kraft pulp. However, in order to simulate other processes such as Lo-solid kraft pulping (pulping at lower temperature) and RDH (rapid displacement heating) some modification is required in order to allow interchange of the liquor during the pulping process (Gullichsen *et al.*,1999; Van Tran, 2005).
- Higher Kappa number pulp > 70, did not produce a pulp which meets the specification for sack Kraft paper grades as opposed to pulp in with Kappa number in the range of 30-70.

## 6.2 Recommendations

Since the experimental results have revealed that improving pulp yield of *Pinus patula* using conventional Kraft pulping (at a cooking temperature of 170 ℃ and liquor charge of 16%) by increasing Kappa number has a negative effect on pulp strength properties, it is recommended that the following studies should be investigated to optimize pulp yield and strength properties for sack paper and linerboard:

1. **Pulping at lower temperature.** This can be done by simulating Lo-solid Kraft pulping or RDH (Gullichsen *et al.*, 1999; Van Tran, 2005).
2. **Treatment of woodchips with sulphide enriched liquor (Black or Green liquor):** This can be done by using sulphide enriched liquor during the impregnation stage followed by normal Kraft cook starting with pulping to Kappa 70 (Olm, 1996; Bykova *et al.*, 1997; Minja, 1998).
3. **Polysulphide Kraft pulping**: This can be done by polysulphide Kraft pulping to Kappa number range of 70-110 or alternatively polysulphide Kraft pulping to Kappa 110-150 followed by alkaline oxygen delignification (Kleppe and Storebraten, 1985).

4. **Modified refining**: This can be done by pulping to a higher Kappa number followed by alkaline peroxide treatment of the high yield pulps prior to HC refining (Jarkko, 2005).

With regard to optimising strength properties by using the HC refining technique, it is recommended that future studies should use a plate which can operate in a consistency range of 32-40%. This will improve the pulp strength properties, particularly for sack paper (Pagiliarini, 1992, Gurnagul *et al*, 2005). Figure 44 shows typical refiner plates recommended for refining of high yield softwood Kraft pulp (Source: IPT and MATECH EUROPE: 2005). This is the D2A504 model plate with small MDF fillings.

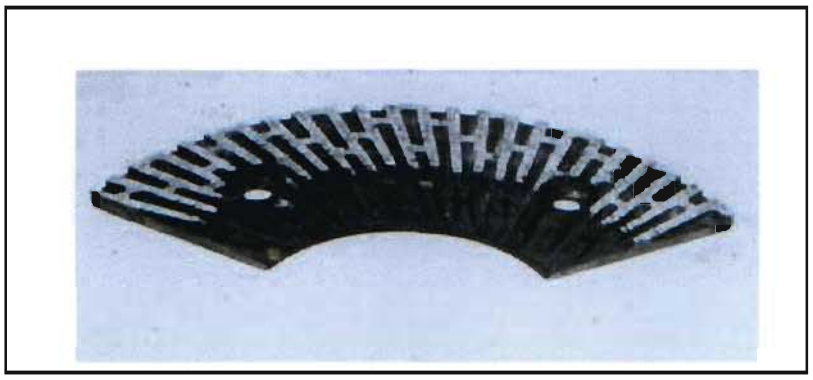


**Figure 44: Typical plate pattern recommended for HC refining (Source: IPT and MATECH EUROPE: 2005)**

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# APPENDICES

## Appendix A: Pulping

### Appendix A-1: Digester input sample calculations

**Wood chip properties**

Wood charge into digester =17 kg

Wood moisture content (%) =13

Water in wood chips: 0.13x17 =2.21kg

Oven dry mass of woodchips: 17- (0.13x17) =14.8kg

**White liquor properties (WL)**

Liquor charge (on oven dry mass of wood) = 16%

Liquor to wood ratio =5:1

E.A = 156.9g/l

Volume of WL charged: $(14.8x0.16x10^3)/156.9=15.1l$

Total volume liquid charged into the digester: 5x14.8= 74l

Make up water to make the liquor to wood ratio:

Total volume of liquid – (Volume of WL +Water in wood chips): 74- (15.1+2.21) =56.7l

### Appendix A-2: H-factor evaluation sample calculation

The temperature changes as cooking cycle progresses. Variation of temperature and time is usually known. The obstacle was to get the relationship between reaction rate constant for Kraft pulping reaction and temperature. Vroom (1957), assumed that the Arrhenius equation $K= e^{-Ea/RT}$ satisfies the rate of delignification for Kraft pulping reaction. Where R is the gas constant, T is the absolute temperature in Kelvin and Ea is the activation energy for the delignification reaction.

$K_r= e^{-Ea/RT}$ ..... (1)

Taking the natural log both sides

$\ln K_r= \ln A- Ea/RT$ ..... (2)

At 100 °C (373K), the equation gives

$\ln K_{100}= \ln A- Ea/373R$ ..... (3)

Subtracting equation 3 from 2, gives

$\ln (K_r/K_{100}) = - Ea/RT + Ea/373R$ ..... (4)

Re-arranging equation 4, gives

$\ln K_r = Ea/R\ (1/373 - 1/T)$ ..... (5)

The Activation energy (Ea) for Kraft pulping has been investigated. Most studies (Vroom, 1957, Christensen, 1998, Kubes *et al*, 2002) have reported an average of 32 kcal/mol (134 kj/mol). The gas constant R is equal to 1.987 cal/gmolK.

Substuting the value of R and Ea in equation 5 gives

$\ln K_r = 32000/1.987\ (1/373 - 1/T)$

$\ln K_r = (43.2 - 16, 113/T)$

$K_r = \ln^{-1}(43.2 - 16, 113/T)$ ..... (6)

From the derivation, equation 6 was used to evaluate the reaction rate constant. For each pulping trial, H-factor was determined (Vroom, 1957):

**H-factor accumulated during the ramp up phase**

After the temperature was reached at 90 °C, the temperature was recorded every 10 minutes until the cooking temperature was reached (170 °C). The calculation of H-factor for the ramp up phase is shown in the Table 14.

**Table 14: H-factor accumulated during the ramp phase**

| Time (Min) | Temperature (°C) | Relative rate | (Average relative rate) x time | Accumulated H[a] |
|---|---|---|---|---|
| 0 | 100 | 1.0 | | |
| 10 | 122 | 11.1 | 6.1x 0.17 | 1.017 |
| 20 | 136 | 44.8 | 28 x 0.17 | 4.76 |
| 30 | 145 | 105 | 75 x 0.17 | 12.75 |
| 40 | 156 | 281 | 193 x 0.17 | 32.81 |
| 50 | 160 | 397 | 339 x 0.17 | 57.63 |
| 60 | 166 | 662 | 532 x 0.17 | 90.44 |
| 65 | 170 | 921 | 792x 0.138 | 109 |
| **Total ramp phase accumulated H-factor** | | | | **283.7** |

**H-factor accumulated during cooking phase**

(Relative rate constant at 170 °C) x holding time at cooking temperature (hrs)

**= 921 x 15/60**

**= 230.25**

**Total H-factor for the given cooking cycle time**

Ramp phase H-factor + cooking phase H-factor

= 283.7 + 230.3

= 514

This H- factor calculation procedure was used for all pulping trials, the results are tabulated in Table 15.

**Table 15: An average total H-factor for given cooking cycle time**

| Cooking time (Min) | Ramp phase Mean H-factor | Cooking phase Mean H-factor | Total cycle time Mean H-factor |
|---|---|---|---|
| 15 | 283.7 | 230.3 | 514 |
| 25 | 284.2 | 383.8 | 668 |
| 35 | 283.7 | 537.3 | 821 |

## Appendix A-3: Calibration of temperature profile

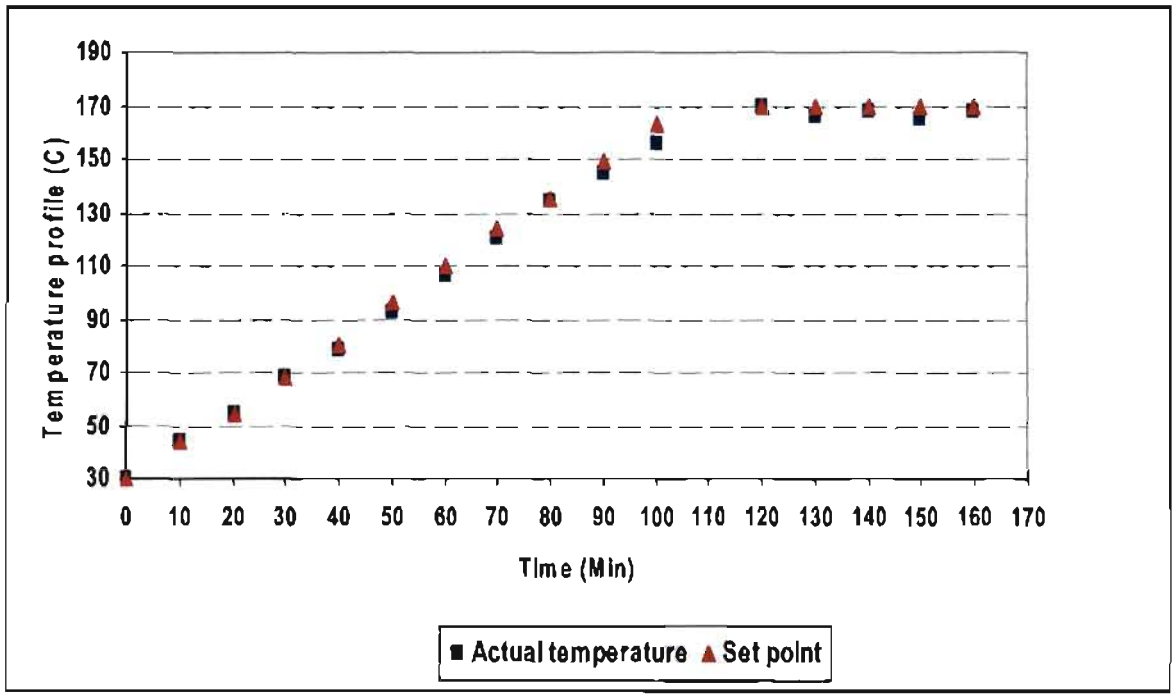


**Figure 45: Digester operation temperature calibration**

## Appendix A-4: Pulping raw data

**Table 16: Cooking liquor specifications average values**

| Composition of cooking liquor | Series of analysis and standardization | | | |
|---|---|---|---|---|
| | *1* | *2* | *3* | *4* |
| Total alkalinity ( g/l ) | 170 | 165 | 168 | 179 |
| Effective alkali ( g/l ) | 156 | 160 | 156 | 154.6 |
| Sulphidity (% ) | 31.8 | 31 | 32.8 | 32.4 |

**Table 17: Pulping results**

| H-factor | | Kappa number | | Shives content (%) | | Total yield (%) | |
|---|---|---|---|---|---|---|---|
| Mean | STD (s) | Mean | STD (s) | Mean | STD (s ) | Mean | STD ( s ) |
| 821 | 3.2 | 100 | 1 | 28 | 2 | 58 | 0.1 |
| 668 | 1.8 | 115 | 1.8 | 44 | 3 | 60 | 0.18 |
| 514 | 3 | 130 | 2.4 | 47 | 2.4 | 61.1 | 0.15 |

**Table 18: Black liquor analysis results**

| Kappa number | Residual alkali (g/l) | |
|---|---|---|
| | Mean | STD (s) |
| 100 | 6.3 | 0.9 |
| 115 | 6.1 | 0.3 |
| 130 | 6.8 | 0.6 |

**Table 19: Pulp pH results**

| Kappa number | Pulp pH | |
|---|---|---|
| | Mean | STD (s) |
| 100 | 9 | 0.3 |
| 115 | 9.2 | 0.6 |
| 130 | 9 | 0.7 |

## Appendix B: Refining

### Appendix B-1: optimisation of HC refiner operating conditions

#### Pulp feeding system

It was anticipated that after filling the hopper with pulp, the pulp would flow to the refining zone, using a vibrating feeder followed by a screw feeder. However, using this set up the pulp was hanging in the hopper and the pulp flow was uncontrollable. Thus it was not possible to quantify the pulp flow rate. The pulp was also not feeding into the refining zone continuously. In consultation with Ideal Patternmakers and Tooling (IPT), a modified screw feeder with a gearbox was installed on the refiner. The arrangement used to transfer the pulp to the refining zone is shown in Figure 46.

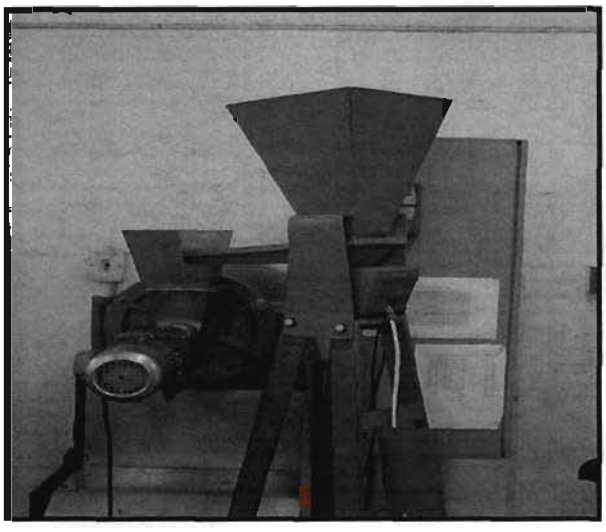

**Figure 46: Refiner after modification on pulp feeding system**

#### Refiner operating speed

The refiner operating speed was 1225 rpm. During the trial run, the grooves in the plates were clogging with pulp leading to jamming of the screw feeder. In consultation with Andritz, it was found that the refiner operating speed was too low. HC refiners operate at high speeds (at least about 3000 rpm) in order to ensure that high centrifugal forces are created to force the pulp outwards from the discs. Thus the pulley and V-belts system were changed. By doing this, the operating speed was increased to 3149 rpm. The operating speed of the refiner was verified by using a tachometer. To enhance proper pulp feeding to the refining zone, the flinger nut with a shape resembling a centrifugal pump impeller was

installed on the rotor plate. Figure 47 shows typical problem was experienced before the modification of refiner speed and installation of a flinger nut.

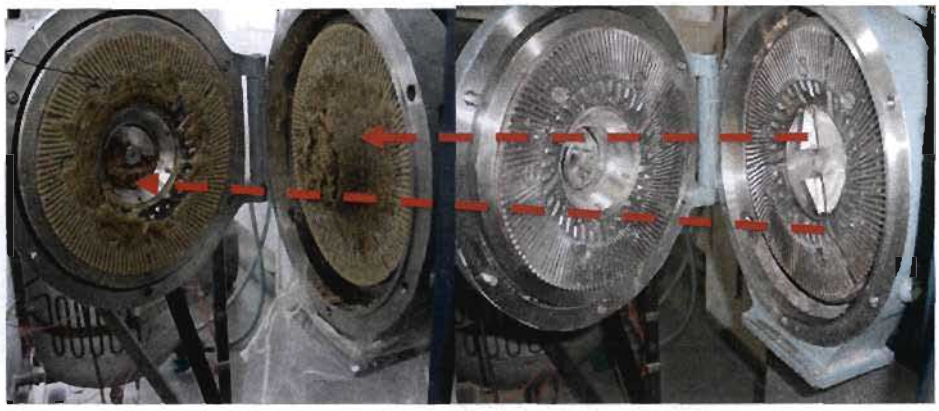

**Figure 47: Effect of refiner operating at low speed and refiner plate without proper flinger nut, evidence of plate plugging and consequent screw feeder jamming**

**Tramming the mill**

The refiner rig was sent to IPT to check the plate parallelism. This was aimed to ensure that the faces of the opposing rotor and stator bars are parallel to each. In addition to that grinding code (bar width, groove width and depth) was verified.

**Trial operating consistency**

The model of the plate pattern used was 2A501, this refiner plate was designed to handle HC pulps of up to 25% consistency (Source: Andritz South Africa). The trial required the feeding of pulp up to 35% consistency. A series of trials were conducted to determine the optimum operating consistency. It was found that it was possible to operate up to 32% consistency without plate clogging.

**Appendix B-2: Calculation for cutting edge length**

The refining action depends on the leading edges. The length of the bar edges that are active against each other per unit of time is referred as cutting edge length (CEL). CEL determines the cutting speed of a particular plate pattern (Lumiainnen, 1995). For each set of refiner plates it was verified as follows:

The bar length were measured using a tape measure

Cutting speed for each refiner plate (m/rev) = total number of stator bars total number of rotor bars x corresponding bar length,

Plate cutting edge length=total cutting speed x refiner operating speed

The HC plates consist of three segments for each plate. The details for HC plates is summarized in Table 20 and one of HC plate segment is illustrated in Figure 48.

**Figure 48: Refiner plate segments used for HC refining**

**Table 20: Calculation data for CEL for HC plates**

| | Total number of bars for the three plate segment | | | |
|---|---|---|---|---|
| **Series no:** | **Stator** | **Rotor** | **Bar length (m)** | **Cutting speed (m/rev)** |
| 1 | 1x3 | 1x3 | 0.35 | 3.15 |
| 2 | 6x3 | 6x3 | 0.53 | 171.72 |
| 3 | 2x3 | 2x3 | 0.30 | 10.8 |
| 4 | 1x3 | 1x3 | 0.35 | 3.15 |
| 5 | 1x3 | 1x3 | 0.40 | 3.6 |
| 6 | 20x3 | 20x3 | 0.55 | 1980 |
| 7 | 4x3 | 4x3 | 0.54 | 77.76 |
| 8 | 1x3 | 1x3 | 0.32 | 2.88 |
| 9 | 1x3 | 1x3 | 0.33 | 2.97 |
| 10 | 1x3 | 1x3 | 0.37 | 3.30 |
| 11 | 1x3 | 1x3 | 0.45 | 4.05 |
| 12 | 1x3 | 1x3 | 0.31 | 2.70 |
| | | Total | | 2266.06 |

Plate cutting edge length=total cutting speed x refiner operating speed

=2.27 km/revx3149 rev/m x 1 m/60s

=119 km/s

The LC plates consist of a single segment (Figure 49). The details for LC plates are summarized in Table 21.

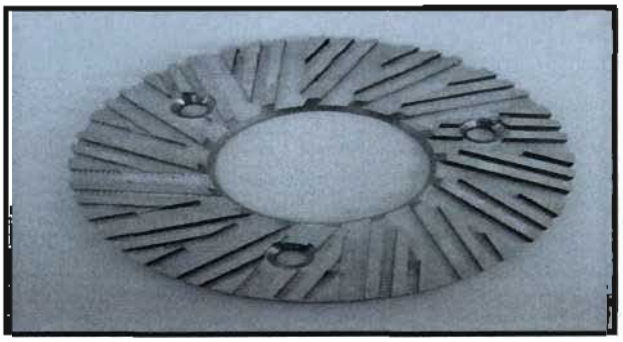

**Figure 49: Refiner plate segments used for LC refining**

**Table 21: Calculation data for CEL for LC plates**

| | Total number of bars for each plate segment | | | |
|---|---|---|---|---|
| **Series no:** | **Stator** | **Rotor** | **Bar length (m)** | **Cutting speed (m/rev)** |
| 1 | 11 | 11 | 0.55 | 66.55 |
| 2 | 10 | 10 | 0.5 | 50 |
| 3 | 16 | 16 | 0.21 | 53.76 |
| 4 | 1 | 1 | 0.26 | 0.26 |
| 5 | 1 | 1 | 0.13 | 0.13 |
| 6 | 1 | 1 | 0.23 | 0.23 |
| 7 | 1 | 1 | 0.11 | 0.11 |
| 8 | 2 | 2 | 0.56 | 2.12 |
| 9 | 1 | 1 | 0.54 | 0.54 |
| 10 | 1 | 1 | 0.26 | 0.26 |
| 11 | 4 | 4 | 0.49 | 6.86 |
| 12 | 1 | 1 | 0.14 | 0.14 |
| | | Total | | 182.06 |

Plate cutting edge length=total cutting speed x refiner operating speed

=0.18206 km/revx1500 rev/m x 1 m/60 s

=4.6 km/s

**Appendix B-3: Refiner operating variable calculation**

Specific refining energy (SRE) is calculated as following (Karnis *et al.*, 1991, Gurnagul *et al.*, 2005):

$P_t - P_o = P_{net}$ ..... (7)

Where

$P_t$ –Total power (measured by $I_t$ vcos F ) or recorded by an integrating instruments

$P_o$ –No load power -Mechanical loss (measured by $I_O$vcosF ) or recorded by an Integrating instruments.

$I_O$ –no load current (amps)

$I_t$ - Total current (Amps) when the refiner operated with load

V-voltage (Watts)

CosF –Phase angle

*SRE* (kWh/t) = $P_{net}$ / Mass flow rate (Bone dry metric tonnes/hr) ..... **(8)**

Where

$P_{net}$ = Net power

Equation 8 was used to evaluate SRE for both HC and LC refining trials.

**HC refiner trial - SRE calculation:**

Refiner operating variables:

Refiner capacity =37 Kw

Power consumption = Power reading (%) x refiner capacity

No load power = 2.18 kW

Consistency (%) =32

Flow rate (kg/hr) = 14.63

Since

Bone dry mass flow rate= refining consistency x pulp mass flow rate

Bone dry pulp mass flow rate (tons/hr)

= 0.32x14.63 kg/hr x$10^{-3}$ ton/1kg

=0.00468 tons/hr

**Table 22: HC refiner trial SRE calculation data**

| Gap size (micron) | Average Gross power (kW) | Average No load power (kW) | Average Net power (kW) | Average Pulp flow rate (bd tons/hr) | Average Target SRE kWh/t |
|---|---|---|---|---|---|
| 625 | 2.84 | 2.18 | 0.66 | 0.00468 | 140 |
| 500 | 2.93 | 2.18 | 0.75 | 0.00468 | 160 |
| 375 | 3.12 | 2.18 | 0.94 | 0.00468 | 200 |
| 350 | 3.30 | 2.18 | 1.12 | 0.00468 | 240 |

**LC Refiner trial - SRE calculation**

Refiner operating variables:

Refiner capacity =22 Kw

Power consumption = Power reading (%) x refiner capacity

No load power (14%) = 3.08 kW

Consistency (%) =4 (Equivalent to 40 kg/$m^3$)

Flow rate (l/s) = 1.5

Since

Mass flow rate= Density x volumetric flow rate

Pulp mass flow rate (tons/hr)

=40 kg/$m^3$ x1.5 l/s x$10^{-3}$ $m^3$/ 1l x 3600 s/1hr x$10^{-3}$ ton/1kg

=0.216 tons/hr

Given that

Target SRE 80, 120,180 and 200 kWh/t

Since

SRE = N.P/pulp mass flow rate

SRE X pulp mass flow rate = N.P, gives net power for the desired SRE

80x0.216 = 17.3 kW

120x0.216=25.9 kW

180x0.216=38.9 kW

200x0.216=43.2 kW

**LC refining consistency determination:**

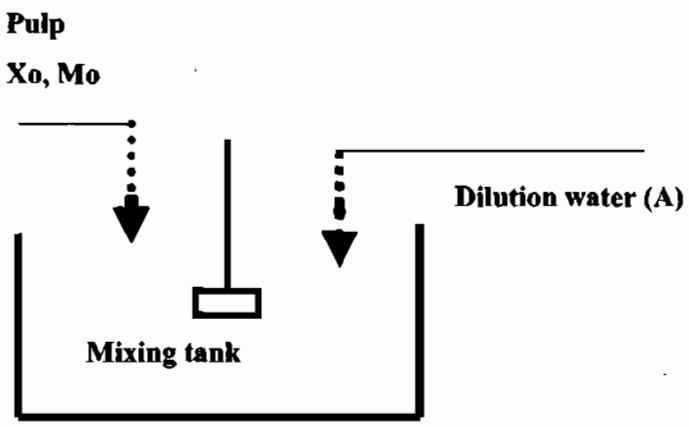


**Figure 50: Low consistency mixing chest**

Given that:

Xo = Initial consistency of wet pulp (%)

Mo = Mass of wet pulp (kg)

$X_1$ = Consistency after mixing of pulp (%)

$M_1$ = Mass of pulp suspension after mixing

A = Amount of water required for dilution to the desired consistency (kg)

Mass balance around the mixing tank

Solid balance

$Xo \cdot Mo = X_1 \cdot M_1$

$M_1 = (Xo \cdot Mo)/ X_1$ .....(9)

Water balance

$A + (1- Xo). Mo = (1- X_1). M_1$ ..... (10)

Substituting equation 9 into equation 10, gives

$A = M_o (xo - x_1)/x_1$ .....(11)

From the refining raw data shown in the table below. The total number of refining stages which gives corresponding net power was evaluated.

Note ** denotes the number of refining stages.

**Table 23: LC refiner trial SRE calculation data**

| Refining stage | Avg Power reading (%) | Avg actual Power (kW) | Avg Net power (kW) | Avg SRE (kWh/t) |
|---|---|---|---|---|
| 1 | 27 | 5.94 | 2.86 | 13.24 |
| 2 | 26.8 | 5.90 | 2.82 | 13.06 |
| 3 | 26.6 | 5.85 | 2.77 | 12.82 |
| 4 | 26 | 5.72 | 2.64 | 12.22 |
| 5 | 26 | 5.72 | 2.64 | 12.22 |
| 6 | 26 | 5.72 | 2.64 | 12.22 |
| **7 | 26 | 5.72 | 2.64 | 12.22 |
| 8 | 25.6 | 5.63 | 2.55 | 11.81 |
| 9 | 25.5 | 5.61 | 2.53 | 11.71 |
| **10 | 25 | 5.5 | 2.42 | 11.2 |
| 11 | 24.8 | 5.46 | 2.38 | 11 |
| 12 | 24.6 | 5.41 | 2.33 | 10.78 |
| 13 | 24.5 | 5.39 | 2.31 | 10.70 |
| 14 | 24.2 | 5.32 | 2.24 | 10.4 |
| 15 | 24 | 5.28 | 2.2 | 10.2 |
| 16 | 24 | 5.28 | 2.2 | 10.2 |
| **17 | 24 | 5.28 | 2.2 | 10.2 |
| 18 | 24 | 5.28 | 2.2 | 10.2 |
| 19 | 24 | 5.28 | 2.2 | 10.2 |
| 20 | 24 | 5.28 | 2.2 | 10.2 |

**HC – RI calculation**

According to Gurnagul et *al.*, (2005), refining intensity for HC refiner can be express as

$$RI = 0.5\left(\frac{\mu_t \times r \times \omega^3}{N \times \mu_r \times C}\right) \quad \text{..... (12)}$$

where,

$r$= radius of the refining zone

N=the number of bars per unit arc length

$\mu_t$ , $\mu_r$ tangential and radial friction coefficients respectively between the pulp and the Plates (Constants specified by manufacture)

$\omega$= Refiner operating speed (radians per seconds)

$C$= Refiner operating consistency

From HC laboratory refiner

$r$ = 0.105m

N= 38

$\mu_c$= 0.75

$\mu_r$ = 0.25

$C$ = 32%

$\omega$ = 3149 rpm (329.6 radians/sec)

Therefore,

*RI* (Expressed as energy per impact)

= 1x0.75x0.105x $(329.6)^2$ / 2x38x0.25x0.32

= 1407 radians/ $sec^2$ or $0.14x10^{-2}$ MJ/Kg when expressed as energy per impact

**Appendix B-4: Refining raw data**

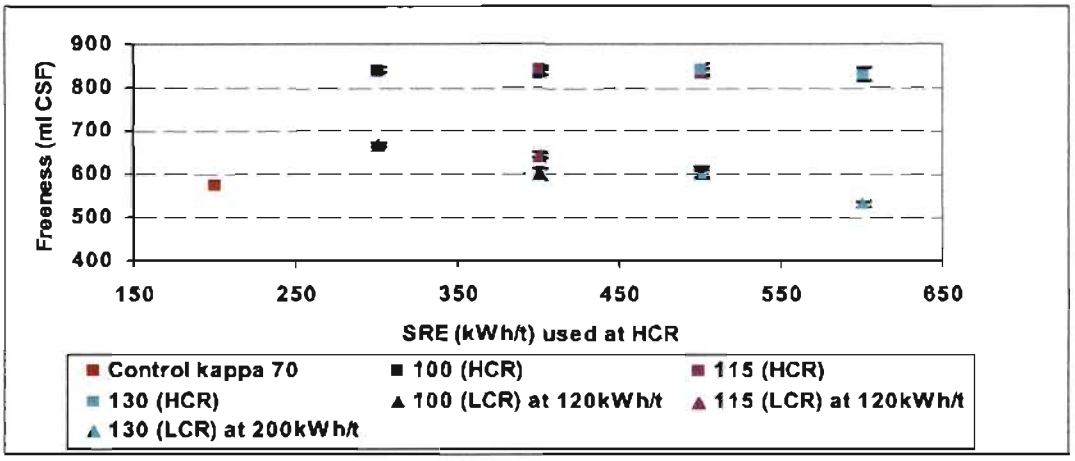


**Figure 51: Graph of freeness vs. SRE**

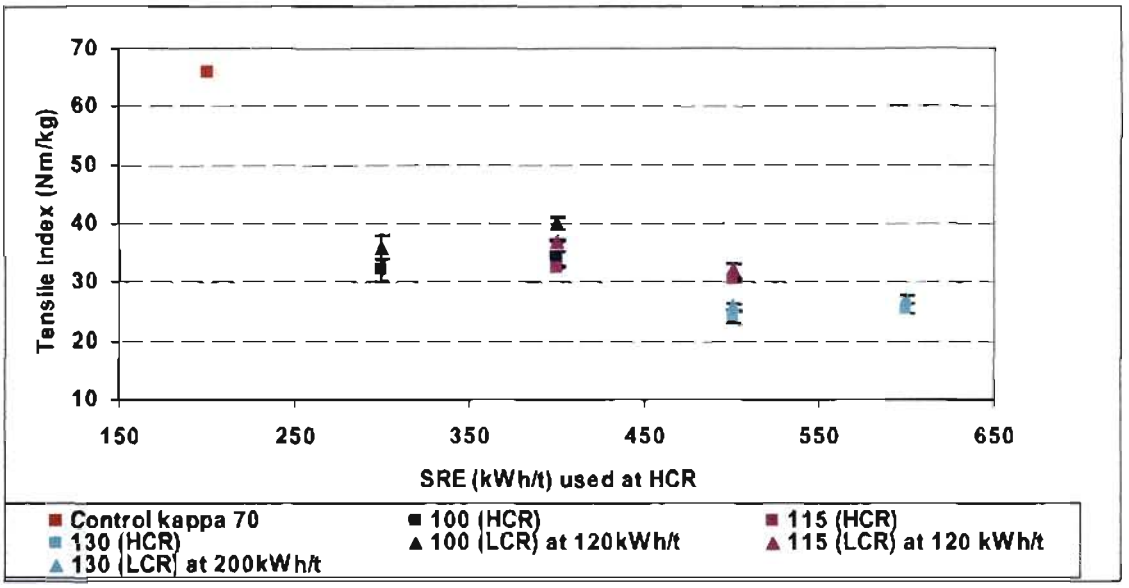


Figure 52: Graph of tensile index vs. SRE

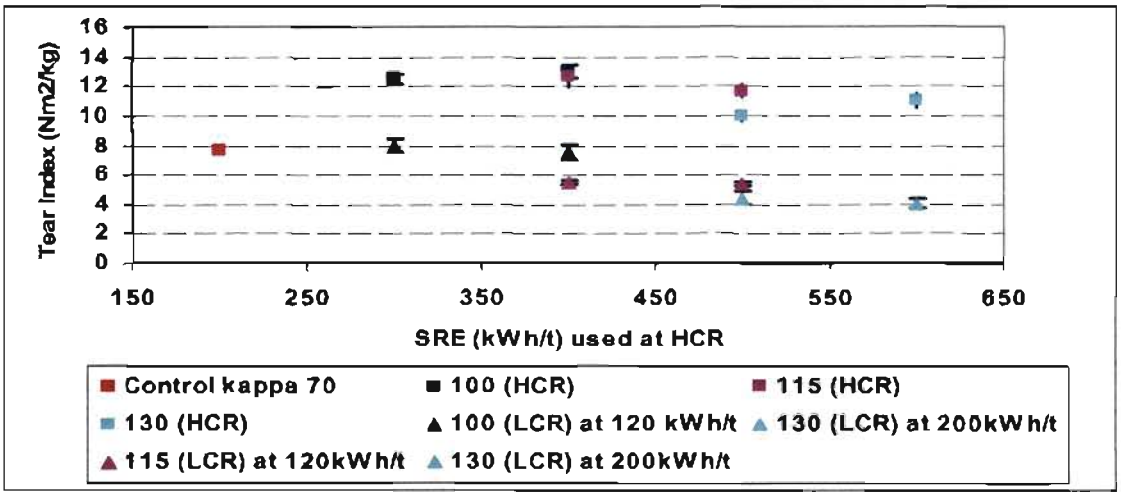


Figure 53: Graph of tear index vs. SRE

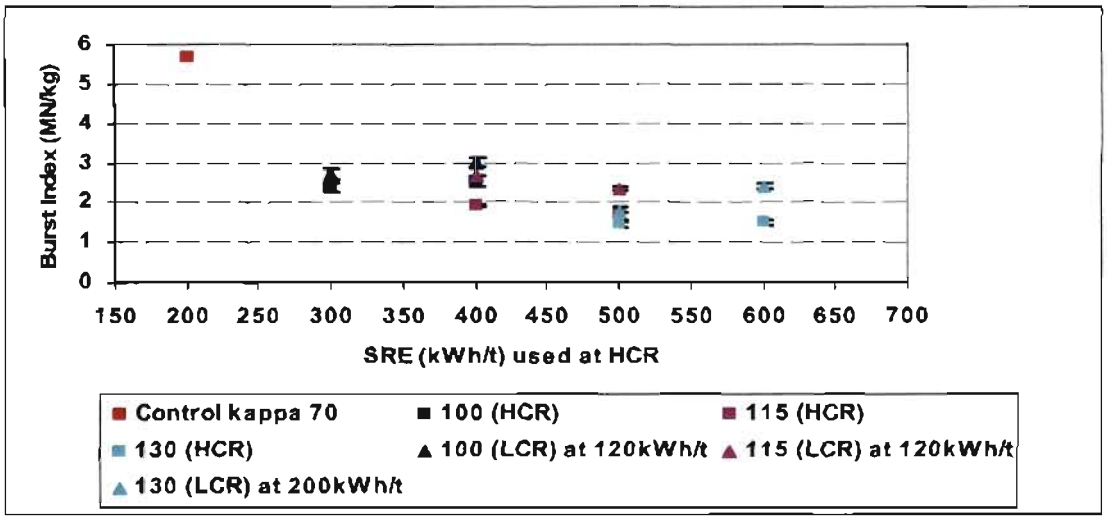


Figure 54: Graph of burst index vs. SRE

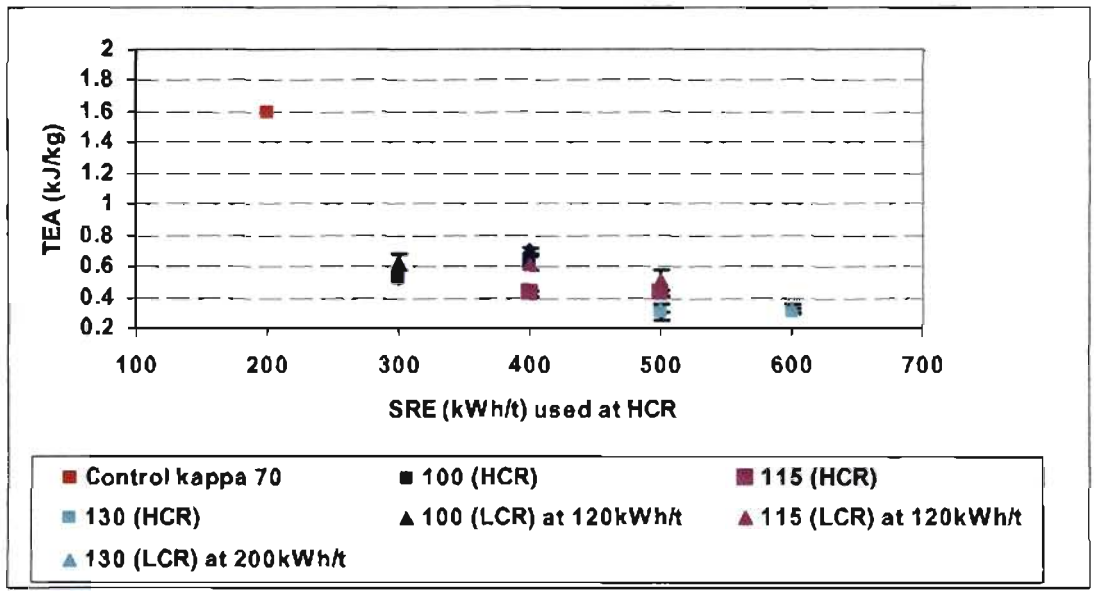


Figure 55: Graph of TEA index vs. SRE

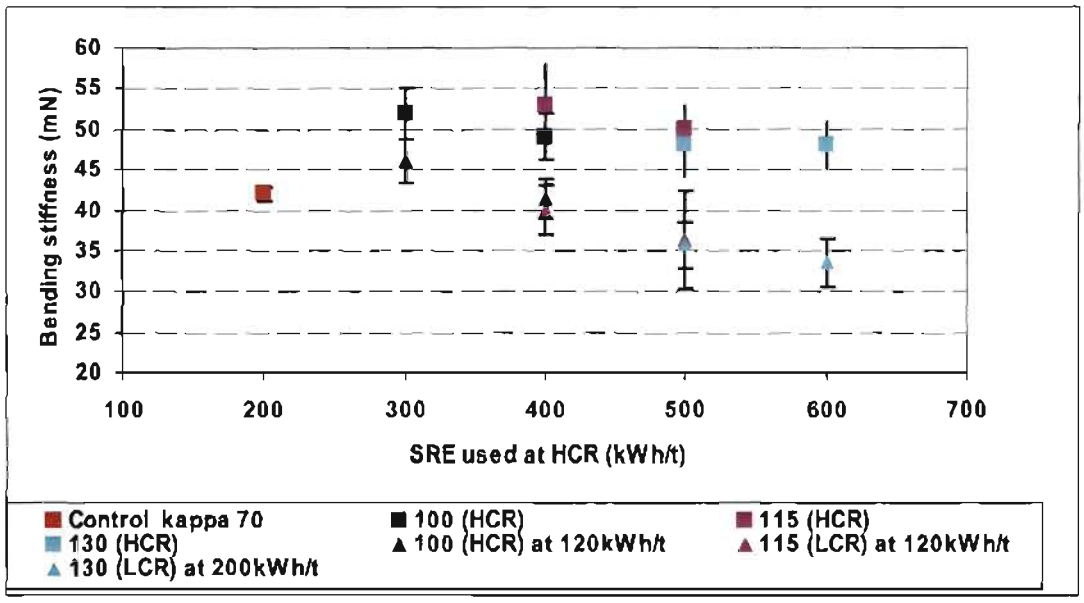


Figure 56: Graph of bending stiffness vs. SRE